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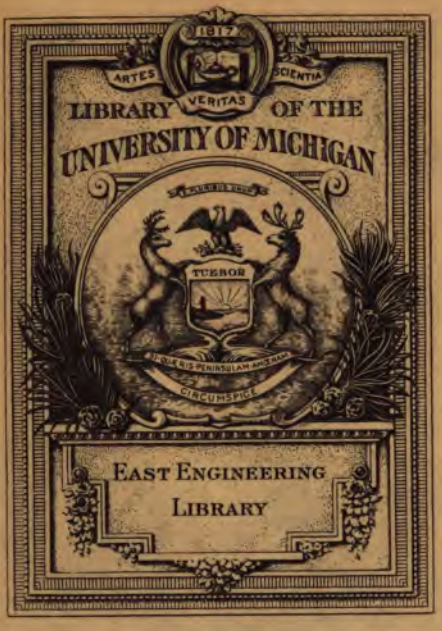
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# INDUSTRIAL CHEMISTRY

BEING A SERIES OF VOLUMES GIVING A COMPREHENSIVE SURVEY OF

# THE CHEMICAL INDUSTRIES


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# THE CARBOHYDRATES AND ALCOHOL

BY
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AND ASSOCIATES

NEW YORK
D. VAN NOSTRAND COMPANY
EIGHT WARREN STREET
1920

PRINTED IN GREAT BRITAIN

# GENERAL PREFACE

THE rapid development of Applied Chemistry in recent years has brought about a revolution in all branches of technology. This growth has been accelerated during the war, and the British Empire has now an opportunity of increasing its industrial output by the application of this knowledge to the raw materials available in the different parts of the world. The subject in this series of handbooks will be treated from the chemical rather than the engineering standpoint. The industrial aspect will also be more prominent than that of the laboratory. Each volume will be complete in itself, and will give a general survey of the industry, showing how chemical principles have been applied and have affected manufacture. The influence of new inventions on the development of the industry will be shown, as also the effect of industrial requirements in stimulating invention. Historical notes will be a feature in dealing with the different branches of the subject, but they will be kept within moderate limits. Present tendencies and possible future developments will have attention, and some space will be devoted to a comparison of industrial methods and progress in the chief producing countries. There will be a general bibliography, and also a select bibliography to follow each section. Statistical information will only be introduced in so far as it serves to illustrate the line of argument.

Each book will be divided into sections instead of chapters, and the sections will deal with separate branches of the subject in the manner of a special article or monograph. An attempt will, in fact, be made to get away from



366121

the orthodox textbook manner, not only to make the treatment original, but also to appeal to the very large class of readers already possessing good textbooks, of which there are quite sufficient. The books should also be found useful by men of affairs having no special technical knowledge, but who may require from time to time to refer to technical matters in a book of moderate compass, with references to the large standard works for fuller details on special points if required.

To the advanced student the books should be especially valuable. His mind is often crammed with the hard facts and details of his subject which crowd out the power of realizing the industry as a whole. These books are intended to remedy such a state of affairs. While recapitulating the essential basic facts, they will aim at presenting the reality of the living industry. It has long been a drawback of our technical education that the college graduate, on commencing his industrial career, is positively handicapped by his academic knowledge because of his lack of information on current industrial conditions. A book giving a comprehensive survey of the industry can be of very material assistance to the student as an adjunct to his ordinary textbooks, and this is one of the chief objects of the present series. Those actually engaged in the industry who have specialized in rather narrow limits will probably find these books more readable than the larger textbooks when they wish to refresh their memories in regard to branches of the subject with which they are not immediately concerned.

The volume will also serve as a guide to the standard literature of the subject, and prove of value to the consultant, so that, having obtained a comprehensive view of the whole industry, he can go at once to the proper authorities for more elaborate information on special points, and thus save a couple of days spent in hunting through the libraries of scientific societies.

As far as this country is concerned, it is believed that the general scheme of this series of handbooks is unique, and it is confidently hoped that it will supply mental

munitions for the coming industrial war. I have been fortunate in securing writers for the different volumes who are specially connected with the several departments of Industrial Chemistry, and trust that the whole series will contribute to the further development of applied chemistry throughout the Empire.

SAMUEL RIDEAL.

# AUTHOR'S PREFACE

THE industries briefly summarized in the present volume in their modern applications have reached a considerable magnitude and involve considerations of an economic character which must be based upon accurate chemical data for future development. A student making a choice of industrial chemical work is apt to think that the industries dealing with starch, sugar, and alcohol, do not afford much scope for the application of chemical principles; but this book may serve to dispel such an erroneous view, and show that not only is a fairly wide knowledge of the chemical constitution of these sugars and their synthesis, formation in the plant and in the laboratory important, but that their decomposition products involve biochemical changes of great interest which cannot fail to be of value in establishing new world conditions of exchange.

Many of the problems specially appeal to the British public, and afford considerations of tropical agriculture and chemical manufacture under economic conditions of power and labour which may modify future progress. It is impossible within the scope of a single volume more than to glance at these varied developments, but it is hoped that the outline here presented may be of interest to many readers, and stimulate greater interest in this country in some of the industries which hitherto as a nation we have not taken into sufficient account. It has been difficult to avoid

references to companion volumes in the series seeing that wood and cellulose supply methyl alcohol and sugars, that fuel production and utilization includes the problem of industrial alcohol, and that the Carbohydrates are still industrially plant products.

SAMUEL RIDEAL.

*July*, 1920.

# CONTENTS

## INTRODUCTION.

PAGE

Cellulose as a natural product, an industrial material, and a chemical substance. General properties of cellulose. Its growth and decay. The production of cotton. Starch. Origin and natural characters. General and chemical properties. Starch paste and gelatinization, soluble starch. Proximate constituents of starch, hydrolysis by acids and enzymes. Dextrins. Sugars. General constitution of sugars. Properties of glucose, fructose, invert sugar, galactose, sucrose, maltose, lactose, raffinose, dextrin . . . . . 1–15

References . . . . . 15

---

## PART I.

# STARCH AND ITS PRODUCTS.

## SECTION I.—STARCH.

Raw materials for the preparation of starch . . . . . 16

Yields of starch from various sources . . . . . 16

(*a*) Wheat starch. Structure of the wheat grain. Preparation by the fermentation process. The sweet process. Gluten . . . . . 17

(*b*) Maize or corn starch. Preparation. Utilization of residues. Corn oil, Germ cake, Chemicalled and unchemicalled starch. Thin boiling and thick boiling starch . . . . . 20

(*c*) Rice starch. Composition of raw material. Softening, grinding, sieving, moulding, drying. Uses of rice starch . . . . . 22

(*d*) Potato starch. Composition of potatoes. Determination of starch content of potatoes. Washing, rasping, sieving, drying. Treatment of residues . . . . . 24

(*e*) Arrowroot, sago, tapioca . . . . . 29

Preparation of soluble starch. Uses . . . . . 29

Uses of starch . . . . . 30

References . . . . . 43

## SECTION II.—DEXTRIN.

PAGE

Properties. Preparation of solid dextrin or British gum. Roasting or Torrefaction process. The acid process . . . . . 31
References . . . . . 43

## SECTION III.—GLUCOSE.

Manufacture of glucose syrup and grape sugar . . . . . 34
Conversion of starch. Neutralization. Filtration. Evaporation . . 36
Filtration through animal charcoal . . . . . 38
Concentration in the vacuum pan. Uses . . . . . 39
Chemical Synthesis of sugars . . . . . 41
References . . . . . 43

## SECTION IV.—MALTOSE.

Industrial preparation of maltose syrup . . . . . 42
References . . . . . 43

# PART II.

# SUGAR.

## SECTION I.—CANE SUGAR.

*The Sugar Cane.* Origin and distribution . . . . . 45
*The Plant.* Cultivation. Soil and water requirements. Composition of the cane. Varieties. Pests and diseases . . . . . 48
*Extraction of the Juice.* Cane mills. Maceration and Imbibition. Megass. Multiple crushing. The diffusion process. Yields. Character of the juice 53
*Clarification.* Defecation agents. Defecators. Sulphuring. Eliminators. Carbonation. Treatment of scums. Filtering media . . . 64
*Evaporation.* Earlier methods. The copper-wall battery. Panela train and concretor. Film evaporators. Vacuum pan, triple effect. Lillie and Kestner evaporators. . . . . 71
*Boiling.* Crystallization. Muscovado tanks. Vacuum pan . . . 77
*Curing.* Crystallizers. Centrifugals . . . . . 78
*Molasses.* Composition. Utilization . . . . . 80
References . . . . . 82

## SECTION II.—BEET SUGAR.

*The Beetroot.* Cultivation. Improvement by selection. Composition . 83
*Manufacture.* Brief history of the foundation of the beetroot sugar manufacture. Early methods of the extraction of the juice. Pressing the

PAGE

rasped pulp. Cold and hot maceration processes. The diffusion process. Theory of the diffusion process . . . . . 87

*Extraction of the Juice.* Washing the roots. Beet slicing machines. Diffusers. Working of the battery. Exhausted pulp. Pressing and drying the pulp. Molasses fodder . . . . . 94

*Purification of the Juice.* Defecation. Single and double carbonation. Sulphiting. Filtration. Treatment of scums. Lime kilns, carbon dioxide. Sulphur burner . . . . . 100

*Evaporation.* Multiple effect evaporators . . . . . 109

*Boiling.* The vacuum pan. Boiling to grain. Control of the boiling . 113

*Treatment of the Massecuite.* Centrifugal treatment of the first massecuite. Crystallization in motion of the second massecuite. Molasses . . 118

*Recovery of Sugar from Molasses.* Osmose process. Elution process. The substitution and separation processes. The strontia process . . 122

References . . . . . 125

SECTION III.—SUGAR REFINING.

Raw sugars. Preliminary treatment or affination. Melting. Filtration. Filtration through animal charcoal. Revivication of char. Boiling to grain. Drying the massecuite. Granulated sugar. Cube sugar. Preparation of invert sugar and saccharum. Table syrups. Refinery molasses 127

References . . . . . 132

SECTION IV.—MINOR SOURCES OF SUGAR.

Sorghum sugar and syrup . . . . . 134

Maple sugar and syrup . . . . . 135

Palm sugar . . . . . 136

References . . . . . 137

SECTION V.—CARAMEL.

Properties. Manufacture. Uses . . . . . 138

References . . . . . 139

PART III.

ALCOHOLIC FERMENTATION.—BEER.

SECTION I.—MALTING.

Barley, malt, diastase. Supplementary materials. Preparation of grist . 140

SECTION II.—MASHING.

Nature of the changes during mashing. Influence of water used for brewing 145

## SECTION III.—BOILING AND HOPPING.

PAGE
The chemical constituents of hops. Cooling and refrigerating . . . 148

## SECTION IV.—FERMENTATION.

Yeasts. The chemical changes during fermentation. High and low yeasts. Pure cultures. Aeration, temperature, duration. Density of wort and attenuation. Baker's yeast. Yeast food product. Diseases of beer. Zymase . . . . . 151
References . . . . . 160

# PART IV.

# WINE.

## SECTION I.—GRAPES AND THE VINE.

Pressing and must. Constituents of the must. Marc . . . . . 163

## SECTION II.—FERMENTATION.

Fermentation and slow fermentation. Storage and treatment. Sweet, dry, and effervescing wines, medicated wines and other alcoholic beverages . 165

## SECTION III.—TARTAR.

Lees, argol, tartaric acid . . . . . 168
References . . . . . 169

# PART V.

# DISTILLATION.

## SECTION I.—GRAIN SPIRIT.

Malting and mashing. Fermentation. Distillation. Rectifying. Absolute alcohol. Proof spirit . . . . . 170

## SECTION II.—POTABLE SPIRIT.

Brandy, rum, whiskey, gin, potato spirit, liqueurs and cordials. Physiological action of alcohol, alcohol as a food, mental effects, action on the digestion, action on respiration and circulation of the blood, alcohol and the performance of muscular acts, effect on the body temperature, poison action, longevity . . . . . 178

## SECTION III.—INDUSTRIAL ALCOHOL.

PAGE
Molasses, potatoes, beetroots, denaturants. Alcohol as a fuel . . . 190

## SECTION IV.—SYNTHETIC ALCOHOL.

From wood, starch, peat, carbon . . . . . 194
References . . . . . 198

# PART VI.

# VINEGAR.

## SECTION I.—PREPARATION OF THE WORT.

Fermentation. Acetification. Acetic acid bacteria. Quick vinegar process. Acetates . . . . . 200

## SECTION II.—ACETIFICATION.

Malt vinegar. Wine vinegar. Vinegar from alcohol . . . . . 203

## SECTION III.—ACETIC ACID.

Acetic acid. Formation, synthetic acetic acid, glacial acetic acid. Aromatic vinegar . . . . . 206

## SECTION IV.—ACETONE AND GLYCERINE.

Industrial preparation of acetone, glycerine from molasses . . . 211
References . . . . . 216

INDEX . . . . . 217

# CARBOHYDRATES

## INTRODUCTION

THE great group of the carbohydrates, including cellulose, starch, the sugars and gums, forms a class of compounds of extreme interest, both from their wide distribution, especially in the vegetable kingdom, and for their utility to mankind, furnishing food, clothing, building materials, fuel, paper and explosives, and the source from which wine, spirits, beer and other beverages are prepared.

They are classified on the basis of molecular complexity into—

Monoses or Monosaccharides, as dextrose and levulose;
Saccharobioses or Disaccharides, as cane sugar and maltose;
Saccharotrioses or Trisaccharides, as raffinose; and
Polysaccharides, including starch and cellulose.

**Cellulose.**—Cellulose in its many forms constitutes the basis of the skeletal framework of all plants, supporting and containing the living protoplasm of the vegetable cell. The cell-walls in the early stages of development consist entirely of cellulose, but as the plant grows they become encrusted with other products of growth either mechanically as colouring matters, resins, and other foreign substances, or chemically, as bodies closely allied to cellulose forming the compound celluloses. The latter are considerably less resistant than cellulose proper to the action of alkalis, oxidizing agents, and the halogens.

Cellulose to form the cell-wall is probably deposited as a hydrated colloid formed indirectly from starch. At this

early stage it is much more susceptible to chemical reagents than in its later less hydrated form. The purest form in which it occurs in nature is in cotton and similar seed hairs, and in pith, but even these contain a small proportion of inorganic constituents in such close combination with the tissue that after ignition they retain the form of the original structure.

As prepared from raw fibrous materials such as cotton, flax, and hemp, cellulose is a white, lustrous, more or less translucent substance of organized structure and possessed of a certain hygroscopic character, so that when air-dried, it usually contains from 7 to 9 per cent. of moisture. The question of moisture is of great importance in the textile industry, since the pliability and tensile strength of the threads are greatly affected by their hygroscopic condition.

The elementary composition of pure, dry, ash-free cellulose is: carbon 44·2, hydrogen 6·3, oxygen 49·5 per cent., corresponding to the empirical formula $C_6H_{10}O_5$, and thus accords with the constitution characteristic of the carbohydrates in which the hydrogen and oxygen are present in the same ratio as in water.

Cellulose is insoluble in water and all simple solvents, but is dissolved in cuprammonium hydrate (Schweizer's reagent) from which it is precipitated by acids, some alkali salts, and by sugar, but in a hydrated and modified form. Its general inertness to chemical reagents renders it useful for filtering purposes. Attention may here be directed to its power of retaining considerable quantities of some salts by absorption. For further details we refer the reader to the volume in this series on Wood and Cellulose.

The immense accumulation of vegetable matter in the world as leaves and wood, consisting mainly of cellulose, is removed to re-enter the cycle of life after being broken down by the action of bacteria and enzymes.

Without attempting to make any estimate of the total production of cellulose in the world by vegetable growth, some idea of its magnitude may be formed by considering the case of one particular plant, cotton, the seed hairs of

which form the product in question. Sir Charles W. Macara states that the world's average cotton crop may now be estimated at 20,000,000 bales of 500 lbs. each, or three times the quantity that was produced ten years ago. There are, moreover, 2 lbs. of seed to every 1 lb. of cotton. The hulls form hay for feeding, and oil is obtained from the seed and the residual cake is used for feeding stock.

**Starch.**—The earliest preparation of starch was no doubt made from wheat, and was called amylum by the Greeks, since it was not obtained by grinding in a mill as flour is. Dioscorides states that the best kind of starch flour is obtained from Cretan or Egyptian wheat. The grain was steeped in water to soften it, and then kneaded and washed with water. The husks were next sieved out, and the deposited starch dried on bricks in the sun, since if left moist it soon became sour. Pliny (lib. xviii. c. 7) gives a similar account, and states that starch was discovered at Chios.

Starch is, next to cellulose, the most abundant material found in the vegetable world, and is present in all green plants, in which it occurs as microscopic granules of apparently organized structure. It is absent from the top of the bud and the extremity of rootlets, otherwise it is found in all parts of the plant in varying amounts; in seeds, with the exception of certain oleaginous seeds, and most abundantly in those of the cereals and leguminosæ, in roots, tubers, stems, and leaves. It acts as a reserve material for the needs of the plant, and forms and disappears by the action of diastases secreted by the protoplasm according as the cell juice is rich or poor in sugars. In the green parts of plants it is associated with chlorophyll, which determines its formation by the action of light from carbon dioxide and water. Although it is the first visible product of this photosynthesis, it is probably a polymerized form of simpler bodies. It gradually disappears from the leaves during the dark, but is formed in darkness when a living leaf is floated on a solution of glucose, galactose, fructose, sucrose, maltose, or even glycerine, but not when lactose or raffinose is used.

Pure starch is a white glistening powder, free from taste or smell, not volatile, infusible, uncrystallizable, and insoluble in water and all neutral solvents. It differs from cellulose in being insoluble in cuprammonium oxide. Under the microscope it is seen to consist of minute granules of a concentrically stratified structure, the size and form of the granules being characteristic of the plant from which the starch is derived. The outer layers are denser than those nearer the nucleus or hilum, which appears as a dark spot and generally occupies an excentric position. The granules, when intact, are quite unacted upon by water, owing to the protective action of the outer layer. When this layer is broken, however, water is readily absorbed, the contents of the granule swell considerably, and a small quantity passes into solution. By appropriate treatment the whole of the contents of the granule may be removed, whilst the outer coating is left in the form of extremely thin layers. Treated with a solution of iodine this outer coating gives a dirty yellow colour, whilst the cell contents are coloured an intense blue. This reaction is characteristic of all starches, and is not produced by any other known substance. The interior contents of the granules have been named granulose, and the substance forming the outer layer, starch cellulose. Another view of the matter is that this granulose, or amylo-cellulose, constitutes from 80 to 85 per cent. of the granules, the rest being a mucilaginous substance, amylopectin, to which the viscosity of starch paste is due. But amylo-pectin is probably derived from amylo-cellulose by condensation just as amylo-cellulose is derived from sugars. Solid starch may be regarded as a coagulated substance which in nature has passed from the state of colloidal solution to the solid form, the passage of solid starch to starch-paste and soluble starch being a reverse process to this. Moreover, in the transference of starch by the action of translocation diastase, whereby it is removed in a soluble form for the needs of the plant, instead of being converted into soluble sugar for this end, it may possibly be merely converted to a soluble starch.

When heated with water to a temperature varying according to the origin of the starch, it swells up and forms a paste, which on cooling forms a jelly. The viscosity of starch paste varies widely, depending not only on the variety of starch used, but also on the treatment during preparation in purifying and drying.

When starch is heated with water under pressure to 150° C. it is converted into a modification known as soluble starch. In this form it is soluble in hot water, but separates out on cooling, or on the addition of alcohol, as a white, flocculent amorphous precipitate. Soluble starch may also be prepared by treating starch with hydrochloric acid, with extract of malt, hot glycerol, or a weak solution of caustic soda. Its specific rotation is $[\alpha]D+202$ at 15° C. in a 2·5 to 4·5 per cent. solution.

The action of dilute acids on starch varies with the concentration, temperature, and pressure, resulting in its gradual hydrolysis or saccharification. The same result is obtained on starch paste by various enzymes, the most active of which are the diastase of malted barley, the ptyalin of saliva, and the pancreatic juice ferment (trypsin). The starch is first converted into soluble starch, then into dextrin and maltose, and finally, on prolonged action, into glucose.

**Dextrin.**—Starch gum or dextrin is a gum-like substance soluble in cold water, and precipitated from its solution in an amorphous form by alcohol or by barium hydroxide. The name dextrin was given to it to indicate its dextro-rotatory power. It was first obtained by heating starch, and was found along with glucose when starch was acted upon by diastase or by boiling with dilute acid. It is present in all starchy seeds during germination and in malted grain. During the progressive hydrolysis of starch by diastase a number of dextrines are formed intermediate between starch on the one hand and the final products, maltose and glucose, on the other. These are of gradually diminishing dextro-rotation and of increasing cupric-reducing power, and certain of them give characteristic colour reactions with iodine. Soluble starch or amylo-

dextrine is first formed and is coloured blue by iodine, next erythrodextrine, coloured red by iodine, and subsequently achroo-dextrines, which give no colour.

The starch molecule may be regarded as consisting of four complex dextrin groups connected with a fifth group of similar character, but which is far less readily resolved than the others by diastase and remains as the so-called stable dextrin on hydrolysis. The first action is the breaking up of the complex molecule of starch $[(C_{12}H_{20}O_{10})20]5$ into the stable dextrin $(C_{12}H_{20}O_{10})20$ and four groups of readily hydrolyzable dextrins or amylin groups. Each amylin group gradually hydrolyzes to a series of complex amyloin or malto-dextrin groups containing one or more molecules of maltose, $C_{12}H_{22}O_{11}$, thus: $(C_{12}H_{20}O_{10})20+H_2O=C_{12}H_{22}O_{11}\cdot(C_{12}H_{20}O_{10})19$, and so on, to $(C_{12}H_{20}O_{10})20 + 19H_2O = (C_{12}H_{22}O_{11})19.C_{12}H_{20}O_{10}$. These complex amyloin groups break up into smaller molecular aggregations, which, however, retain all the characteristics of the amyloins, and this goes on until the maltose stage is reached.

An alternative view of the process is that the stable dextrin stage is reached when the whole of the amylose of starch is converted into maltose, and the amylo-pectin, which forms one-fifth of the starch, is left, and this is only slowly converted into maltose by another ferment, dextrinase, also present in diastase.

Commercial dextrin is a light powder or semi-transparent masses generally of yellowish colour with a smell somewhat like new bread. It dissolves completely in water and gives a red colour with iodine. The specific rotation is $[\alpha]D=+195$.

**Sugars.**—Sugar in the popular sense always means cane sugar or sucrose, whether derived from the sugar cane, beetroot, or other plant, and to the manufacturer is known as crystallizable sugar because the glucose and fructose present along with it, especially in cane products, remain behind in the mother syrup or molasses. Unlike starch, which is present as a solid, sugar is found in the plant in solution in the juice of the cells where it is stored as a reserve food material.

The sweet taste characteristic of sugar is possessed by a number of other substances in no way related to it in chemical constitution, but the reason why these substances taste sweet is not known. It is a curious fact that dextrorotatory asparagine has a sweet taste, while that of l-asparagine is disagreeable and cooling. Pasteur suggests that the substance of the nerves dealing with taste behaves as an optically active substance and reacts differently with each acid. Acetate or sugar of lead, the salts of beryllium (glucinum) and glycerine, all have a sweet taste. Of the coal-tar sweetening agents saccharine, o-anhydrosulphamine benzoic acid, or benzoic sulphimide, is five hundred times as sweet as cane sugar. It is often used in the form of its more easily soluble sodium salt. Dulcine or sucrol, mono-p-phenetol carbamide, glucine, amido-triazine sulphonic acid or its sodium salt, and sucramine or methyl saccharine are also intensely sweet, but none of these chemical substances have any nourishing value, while sugar is valuable as a food.

By preparation from natural products or by chemical synthesis monosaccharide sugars have been obtained containing from three to nine atoms of carbon in the molecule, but the sugars dealt with industrially have six atoms of carbon or a multiple of this, namely, twelve atoms in the disaccharides sucrose, maltose, and lactose, and eighteen in the trisaccharide raffinose. The hexoses are usually spoken of industrially as reducing sugars from their action on salts of copper.

**Glucose.**— Dextrose, or dextroglucose, is also called grape sugar from its occurrence in the juice of grapes and other fruits associated with fructose (lævulose); these two hexoses are probably derived from cane sugar, as all three are usually present, and cane sugar is easily resolved into glucose and fructose by hydrolysis whereby a molecule of water is taken up thus—

$$\underset{\text{Cane sugar.}}{C_{12}H_{22}O_{11}} + H_2O = \underset{\text{Glucose.}}{C_6H_{12}O_6} + \underset{\text{Fructose.}}{C_6H_{12}O_6}$$

From the more complex sugars or other carbohydrates it may also be prepared when these are hydrolyzed by

suitable enzymes or by acids, as from maltose or malt sugar, lactose or milk sugar, starch and cellulose.

It crystallizes in cauliflower-like masses of needles from an aqueous solution with one molecule of water of crystallization, but the anhydrous substance may be obtained as needle-shaped crystals from a solution in alcohol. Anhydrous glucose melts at 146° to 147° C. to a colourless glass, but the hydrate has no proper melting-point. When glucose is heated above its melting-point it becomes brown at once; at 170° it loses water and the residue contains glucosan. At higher temperatures it is converted into caramel. Dilute aqueous solutions may be boiled without change, but more concentrated solutions decompose. Ammonia turns a solution of glucose yellow at ordinary temperatures and alkalis generally darken the solutions on heating and the action of oxidizing agents is accelerated in presence of alkalis. The action of acids upon glucose is greatly affected by the concentration. Glucose may be dissolved in cold concentrated sulphuric acid without charring. From the sulphonic acids thus formed dextrins may be separated similar to those obtained from starch. Hydrochloric acid of 7 to 10 per cent. concentration produces more humic acid than sulphuric acid of the same strength. Sodium amalgam reduces glucose to the corresponding hexahydric alcohol, sorbitol, no mannitol being formed if the solution is maintained slightly acid. The reducing action of glucose solutions on metallic salts varies according to the conditions, thus a neutral solution of copper sulphate gives metallic copper, but an alkaline one, as Fehling solution, gives red cuprous oxide. Ammoniacal solutions of gold, silver, and platinum are reduced to metal, giving beautiful mirrors.

Dextroglucose exhibits the phenomenon of mutarotation, formerly called birotation from the fact that a freshly prepared solution has a rotatory power on polarized light about twice as great as that which it eventually assumes on standing for some time. The change may be brought about immediately by adding a few drops of ammonia to its solution. Mutarotation is explained by the existence

of two isomeric closed-chain forms, $\alpha$ glucose with a rotatory power $[\alpha]D+110$ and $\beta$ glucose, $[\alpha]D+19$, the stationary rotation of $[\alpha]D+52{\cdot}5$ of the solution being due to an equilibrium mixture of the two forms, the proportions present depending on the concentration of the solution and on the solvent.

Since the aldohexose $C_6H_{12}O_6$ or $CH_2OH.(CHOH)_4COH$ contains four asymmetric carbon atoms, there are in all sixteen possible stereoisomers, and there is, corresponding to ordinary glucose (d-glucose), a lævorotatory isomeride of equal and opposite rotatory power, l-glucose. Moreover, as two closed-chain forms ($\alpha$ and $\beta$) should exist for each of these, the total number of isomeric aldohexoses is 48, but only three of these occur naturally, namely, d-glucose, d-mannose, and d-galactose.

Five of the six atoms of oxygen in the molecule of glucose are thus to be regarded as present in the alcoholic form as hydroxyl (OH), while the remaining one under certain conditions shows aldehydic functions. Glucose accordingly forms with metallic hydroxides compounds called glucosates, similar to the alcoholates, and also yields esters with acids. Calcium glucosate is more soluble than the corresponding compound with levulose, and this fact is utilized in the preparation of levulose from invert sugar.

The taste of glucose is only about half as sweet as cane sugar.

**Fructose.**—Fructose, formerly called lævulose or fruit sugar, is a keto-hexose, and is now termed d-fructose to indicate its configuration, and without reference to its lævo-rotation. When found free in nature it is almost always associated with glucose as a constituent of plant juices, such as in fruits, the sap, and in the nectar of flowers. In honey the percentage of fructose is slightly greater than that of glucose.

It is formed also by the hydrolysis of inulin, a polysaccharide which takes the place of starch as a reserve material in the roots and tubers of many plants. Among these may be mentioned elecampane (*Inula Helenium*), dahlia.

dandelion, chicory, and the Jerusalem artichoke, the amount present being greatest in autumn. Inulin may be detected in plant tissues by treating a thin section with strong alcohol or glycerol and examining under the microscope when the inulin is observed as sphæro-crystals in the cells.

Fructose may be prepared from invert sugar by treating a 6 to 8 per cent. solution with slaked lime at 30°–35° C., filtering quickly and cooling to 0°. Calcium lævulosate crystallizes out on standing. The crystals are washed with ice-cold water and decomposed with oxalic acid, and the filtrate evaporated in vacuo at a low temperature. The syrup crystallizes much less readily than glucose. Fructose is very soluble in water, but only very slightly in cold alcohol. In hot alcohol it is easily soluble and crystallizes from this solution in very fine needles which are anhydrous.

A solution of fructose is strongly lævorotatory, $[\alpha]D-92$, and the polarization changes considerably with temperature and concentration. It shows mutarotation, a freshly prepared solution shows $[\alpha]D-106$. The syrup readily darkens on heating. Alkalis act upon it similarly to glucose. Sodium amalgam gives equal amounts of mannitol and sorbitol. Fructose as a pharmaceutical preparation is recommended for diabetic patients; its taste is far sweeter than that of cane sugar.

**Invert Sugar.**—Invert sugar is a mixture of equal parts of glucose and fructose, and is found in many plant juices. In unripe canes it is present in considerable amount. It may be prepared from cane sugar by the hydrolyzing action of acids, enzymes, and salts. The rate of inversion by various acids runs closely parallel to their electrical conductivity, and thus appears to be dependent on the ionization of the acids, but the rate is increased by rise of temperature far more than can be accounted for by the increase in ionization or the increased speed of the H ions to which the catalytic action is attributed.

The most important inverting enzyme is invertase, which is present in the leaves, buds, fruit, and reserve organs of

many plants. It may be prepared from yeast by allowing this to undergo autolysis.

Invert is readily fermented. It forms salts with bases. In assessing the value of raw sugars for refining purposes it is assumed that invert sugar renders its own weight of cane sugar uncrystallizable. On heating invert sugar with alkalis dark-coloured products and soluble salts are formed. It reduces alkaline solutions of copper salts. When concentrated it forms a syrup the colour of which is dependent on the purity. When pure the syrup readily becomes pasty from the deposition of crystals of glucose.

Both as a syrup and in the form of a cheese-like paste it is largely used by brewers and termed "saccharum."

**Galactose.**—Galactose is a reducing sugar formed along with glucose on the hydrolysis of lactose or milk sugar. As an anhydride product or galactan it is a constituent of many gums, hemicelluloses, mucilages, and pectins. Agar-agar on hydrolysis by boiling with 2 per cent. sulphuric acid is largely hydrolyzed to d-galactose. It is of interest industrially from its occurrence as a constituent of raffinose which is found in beet sugar. On oxidation it yields mucic acid. It is fermented much more slowly than glucose. d-Galactose is strongly dextrorotatory and shows mutarotation. The initial $[\alpha]D$ is about $+140$, the constant value $+81$. It is less sweet than glucose. It crystallizes in the form of large prismatic needles which contain one molecule of water of crystallization. It is not found free in nature, but the wide prevalence of galactans in fodder plants may provide the source of lactose in the milk of the herbivora.

**Sucrose.**—Sucrose or cane sugar is the best known of the sugars, and is found widely distributed, being found in nearly every part of the plants. Its manufacture, however, is mainly restricted to the sugar cane in the tropics, the beetroot in temperate climates, sorghum, and sugar maple in North America, and a smaller proportion from the date palm in the East.

It crystallizes in anhydrous tables belonging to the monoclinic system, with hemihedral faces. The shape of

the crystals is modified by impurities; the presence of raffinose causes the formation of pointed needles. Its specific gravity at $\frac{15^\circ}{15^\circ}$ C. is 1·591. There is contraction on solution in water, and on dilution, the maximum being reached at 55–56 per cent. At 20° C. 100 grammes of water dissolve 203·9 grammes of sucrose, and at 100°, 487·2 grammes. Sucrose is soluble in 80 parts of boiling absolute alcohol, more easily soluble in dilute alcohol, but insoluble in ether. The solubility in water is increased by the presence of many salts and organic substances. Such impurities play an important part in the sugar industry in preventing the crystallization of sugar and its recovery from molasses. The sugar is supposed to form compounds with the salts, which have then a greater solubility than sucrose alone; such salts are called melassigenic.

The boiling-point of aqueous solutions of sucrose are—

| Per cent. sucrose | 10 | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 90·8 |
|---|---|---|---|---|---|---|---|---|---|
| Boiling-point °C. | 100·4 | 100·6 | 101·0 | 101·5 | 102·0 | 103·0 | 106·5 | 112·0 | 130·0 |

The specific rotation is $[\alpha]D^{20^\circ}+66·5$, and is slightly affected by concentration and temperature. Sucrose is not directly fermentible by yeast, being first hydrolyzed (inverted) by the invertase secreted by the majority of yeasts. Although, finally, the same amount of alcohol and carbonic acid is produced as from glucose and fructose, the process is retarded by the effect of the inversion which must precede fermentation. *Saccharomyces octosporus* and *S. apiculatus*, which do not secrete invertose, do not ferment sucrose. It has been suggested to use such organisms to ferment away the invert sugar present in molasses so as to obtain the sucrose by subsequent crystallization, but the process has not proved a success. Sucrose also undergoes lactic and butyric fermentations. In the viscous fermentation frequently met with in sugar factories the sucrose is converted into the gum, dextran. This result is produced by the organism *Leuconostoc mesenterioides* as well as by other organisms. The masses of gum are spoken of as frog's

spawn. Various *Citromyces* ferment aerated solutions of sucrose with the production of citric acid.

When sugar is heated above its melting-point it begins to lose water and decompose. At 170° to 190° C. caramel is formed. Solutions of sucrose also deteriorate on prolonged heating; in the presence of acids inversion takes place. Sucrose forms compounds with metallic bases, and of these the sucrates of the alkaline earths are of technical importance.

Calcium monosucrate is formed by dissolving finely powdered quicklime in sucrose solutions maintained at a low temperature and precipitating the compound with alcohol, $C_{12}H_{22}O_{11}.CaO+2H_2O$. It is easily soluble in water, and the solution on heating becomes turbid, the monosucrate decomposing into trisucrate and sucrose. $3C_{12}H_{22}O_{11}.CaO=C_{12}H_{22}O_{11}.3CaO+C_{12}H_{22}O_{11}$.

Calcium bisucrate separates in white crystals on cooling with ice a sucrose solution to which two equivalents of quicklime have been added.

Strontium bisucrate begins to separate from a boiling sucrose solution as soon as two equivalents of strontium hydroxide have been added, and the separation is almost complete with three equivalents. The bisucrate is decomposed in cold water into monosucrate and strontium hydroxide. Barium monosucrate is separated in crystalline form on cooling a solution of sucrose containing an excess of barium hydroxide.

**Maltose.**—Maltose, or maltobiose, is obtained by the action of malt diastase on starch paste. The hydrolysis at 50° to 60° C. gives a mixture containing 80·9 per cent. of maltose and 19·1 of dextrine. Lower temperatures give a greater yield, but the action is slower. The hydrolysis with acids converts the maltose into two equivalents of d-glucose. Maltose crystallizes in groups of very fine needles, containing one molecule of water of crystallization. It is very soluble in water, and also soluble in alcohol. It exhibits mutarotation, for ordinary anhydrous maltose $[\alpha]D=+140$. Although a biose, one of the two hexose residues exhibits

aldehydic functions, and consequently it reduces Fehling's solution, but about one-third less than glucose. It does not reduce acetate of copper. Its hydrolysis by acids is only about one-fifth of the rate at which sucrose is hydrolyzed. It is hydrolyzed by the enzyme maltase, but not by invertase. Alkalis act upon it in much the same way as on glucose.

**Lactose.**—Lactose, lactobiose, or milk sugar, is present in the milk of mammals to the extent of 3 to 6 per cent., and may be prepared from whey by boiling to coagulate albuminoids and concentrating the filtrate, using animal charcoal for decolorizing the syrup.

It contains a glucose and galactose residue, and these sugars are formed on hydrolysis. It forms large orthorhombic crystals containing one molecule of water. It exhibits mutarotation, the stable form has $[\alpha]D^{20°}+52{\cdot}5$. It reduces Fehling a little less than glucose, but not the acetate, and is thus distinguished from reducing sugars. Sodium amalgam yields dulcite and mannite. Alkalis act on it as on glucose. Yeast ferments it only with difficulty, but the kefir organism ferments it readily owing to the presence of the ferment lactase which it secretes. The taste of lactose is only faintly sweet.

**Raffinose.**—Raffinose is a trisaccharide, being a condensed product of glucose, fructose, and galactose. It is present in beetroots to the extent of 0·02 per cent., and in cotton seed, 3 per cent.; beetroot molasses may contain as much as 16 per cent. It crystallizes in warty crusts of long monoclinic needles, with 5 molecules of water, and its presence in commercial sugars frequently causes sucrose to form elongated crystals, or spiky crystals as they are termed. It is less soluble in cold water, but more soluble in hot water than sucrose. On heating slowly at 80° to 105° C. it becomes anhydrous, but decomposes if heated quickly. Sugars containing raffinose become brown when heated. It has no action on Fehling's solution, and is only slowly attacked by alkalis. Bottom yeast ferments it completely, but top yeast, which does not contain the ferment melibiase, only ferments the levulose portion, leaving the glucose and

galactose combination, melibiose, unattacked. The rotatory power of raffinose hydrate is $[\alpha]D^{20°}+104{\cdot}5$ without mutarotation. The presence of raffinose in raw beet sugars has therefore a disturbing effect on the polarization for the valuation of these products, and its amount must be determined as far as possible. This is effected by moderate warming with dilute acid, whereby fructose is split off and resulting polarization reduced to $[\alpha]D^{20°}+53{\cdot}5$ for the mixture of fructose and melibiose. Prolonged heating with hydrochloric or sulphuric acid resolves melibiose into d-glucose and d-galactose, the same products of hydrolysis as are obtained from lactose. This hydrolysis is not effected by acetic, tartaric, citric, or lactic acids. Raffinose forms compounds with soda, lime, strontia, and lead oxide.

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# PART I.—STARCH AND ITS PRODUCTS

## SECTION I.—STARCH

STARCH is prepared on the industrial scale from the parts of those plants in which it occurs in greatest abundance. The seeds of the cereals contain starch as their principal ingredient. Wheat contains 55 to 65 per cent.; barley, 38 to 46 per cent.; oats, 28 to 38 per cent.; rye, 44 to 47 per cent.; maize, 54 to 67 per cent.; rice 70 to 76 per cent.; potatoes, 16 to 23 per cent.

Wheat, the original material from which starch was manufactured, is not used so much as formerly. There are several reasons for this, not the least being the desire to retain the expensive wheat for food purposes. The large amount of gluten present, 12 to 16 per cent., renders the process difficult, and the starch being made by souring away the gluten, the vicinity of a wheat starch factory was unpleasant, and became a nuisance to the workmen and the inhabitants of the neighbourhood, so that frequent enactments were necessary to regulate the manufacture. Maize and rice are now mostly employed. The seeds of the leguminosæ also afford about 38 per cent. of starch.

Early in the eighteenth century the potato began to be used as a cheaper source of starch than that of wheat, and potato starch is now made in far greater quantity than other kinds, especially in Germany.

Tous les mois is a variety of starch made from the tubers of *Canna edulis*. Portland sago or Portland arrowroot is obtained from the tubers of *Arum maculatum*. Salep, once largely consumed, and still used in Turkey and the East as a food, is a starch derived from the tubers of various kinds

of *Orchis*. The roots, about 30 pounds in weight, of the bitter cassava, *Manihot utilissima*, a native of South America, contains prussic acid as well as starch, but this poisonous ingredient is removed by washing the grated roots and recovering the tapioca starch after settling. In a similar manner prussic acid contained in haricot beans is removed by soaking the beans in water, which is then poured away, leaving the beans free from it and fit for food. Arrowroot is obtained chiefly from the rhizome or root-stock of *Maranta arundinacea*, a native of the West Indies and Brazil. Sago is likewise a starch mainly produced from the stems of the sago palms, *Sagus rumphii* and *S. levis*, and a coarser kind is obtained from the nuts of *Cycas revoluta* in Ceylon and the East Indies. Banana starch is prepared from the unripe fruits of *Musa sapientium*.

**Structure of the Wheat Grain.**—A grain of wheat consists of three parts, the germ or embryo situated near the base, the endosperm, which forms the bulk of the grain and is formed of a number of thin-walled polygonal cells filled with starch granules embedded in proteins (Fig. 1), including gluten, and the various coverings which constitute the bran. One of these coverings, the aleurone layer, is conspicuous as a single layer of cells, rectangular in section, filled with minute grains of undissolved protein, the aleurone grains or "crystals."

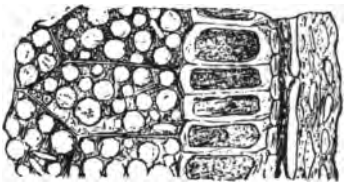

FIG. 1.—Transverse section of Wheat Grain.

Gluten does not exist as such in the grain, but is formed in the presence of water by the interaction of gliadin and glutenin. Of these proteins glutenin is not dissolved by neutral aqueous solutions, by saline solutions, or by alcohol. Gliadin is soluble in 70 per cent. alcohol, is nearly or quite insoluble in water or salt solutions, but readily forms salts with acids or especially with alkalis, and these salts are soluble in water. The aim of the manufacturer of starch is to set free the granules from the cells in which they are

enclosed and to separate the starch from the other constituents.

The average composition of wheat is: starch 68·0, sugar 3·2, cellulose 2·2, fat 1·7, proteins 11·0, mineral matter 1·9, water 12·0 per cent.

**The Fermentation Process.**—Although the sour or fermentation process of preparing wheat starch is wasteful owing to the loss of the valuable constituent gluten, and is now superseded by the sweet or Martin's process, yet a short description may not be out of place considering the chemical changes which occur.

The wheat grains, after a preliminary cleaning, are soaked in water until sufficiently soft to be crushed between the fingers. This is best done in a metal cistern with a conical bottom and provided with a wide perforated pipe in the centre into which water is passed from below. When soft the wheat is either placed in hempen bags and the starchy matter pressed out by treading the bags in water, or the grains are crushed in a mortar mill. The impure starchy liquid is run into tanks where it is allowed to remain for two or three weeks, some liquor from a previous fermentation or sour dough being added, and the mass is stirred occasionally; the liquid is now covered with mould and should have a pleasant vinous odour, but it is difficult to prevent the production of stinking gases. During the fermentation the sugar and some starch acetifies with the production of acetic, propionic, butyric, and lactic acids, and the gluten is thereby acted upon and softened, losing its stickiness, and is partly dissolved, setting free the starch granules. The acid liquor is decanted and run to waste, and the mass washed repeatedly with water so long as this becomes coloured, and then allowed to settle. The various layers are removed and the starch washed through fine sieves to separate particles of bran and fibre. The deposited starch is then placed in shallow boxes lined with canvas, and when compact is turned out, cut into blocks and dried on porous bricks, then stoved, scraped, and packed.

The starch may be more quickly recovered in a

centrifugal separator with a solid drum. The purer starch forms a compact layer at the periphery; the less pure starch milk is run off to form a second quality and fresh liquid run in until a thick layer is obtained. The impure yellow superficial layer is taken off with a sponge. The starch is removed, stirred with water, blued if necessary, and put into suction moulds. In order to avoid the development of mouldiness the starch is dried at once either in the open or in stoves at 30° to 70° C. At a certain stage the cakes shrivel at the surface. This impure starch is scraped off, and the cakes broken into blocks, wrapped in paper and dried. It then acquires the radiating columnar structure which is regarded as a criterion of its good quality. The addition of gluten to potato starch does not enable it to assume a similar structure. Wheat starch is regarded as the best starch for dressing and finishing linen, and the only two wheat starch factories in the United Kingdom are situated in the north of Ireland in the vicinity of the flax industry there.

**The Sweet Process.**—In the preparation of wheat starch by the sweet or non-fermentation process flour is preferably used. This is kneaded with water to a stiff dough and left for a time for the water to permeate thoroughly. It is then washed on a fine sieve under a jet of water until all the starch forms a milk and the gluten is left. This is done on a reciprocating frame with a number of holes in the bottom covered with gauze. The frame bears a number of rollers to assist the operation and is supported in a trough underneath which is an inclined shoot and tanks to collect the starch. When the masses of gluten are removed from the sieve they are placed at the ends of the frame against buffers, and there beaten to remove the last portions of starch. The usual washings and settlings as described above are then proceeded with, but a better result and an increased yield of first quality starch is obtained by treating the impure starch with a weak solution of caustic soda sufficient to give a blue reaction to litmus. After standing some time part of the gluten is dissolved, and the remainder altered so as to be more readily removed on the sieves.

**Gluten.**—Gluten is a valuable digestible protein containing 18 per cent. of nitrogen, and when recovered fresh is used to mix with dough for baking bread, and in other food stuffs. Inferior starches rich in gluten are used in the same way and also as fodder. Gluten does not dry easily, and when wet readily undergoes putrefaction. When allowed to ferment at 50° C. it liquefies, and may then be dried on metal plates to form transparent sheets used as a kind of glue for leather in boot manufacture. Mixed with salt and fashioned into strips and dried, it can be reduced to powder and used as flour.

**Maize or Corn Starch.**—The structure of a grain of maize is similar to that of a grain of wheat already described. The average analysis of maize is: starch 55, other carbohydrates 15, proteins 10·5, fat 5, ash 2·5, water 12 per cent. Of the proteins present one is soluble in water, and the gluten is soluble in dilute caustic alkali.

The maize, after being cleaned from dust, dirt, and other chance impurities, is steeped in water at about 60° C. for 30 hours or less to swell and soften the grain, and a little weak sulphurous acid is added to prevent fermentation or putrefaction, the water being kept in circulation by means of steam siphons. The steep waters are concentrated to be used for feeding cattle. The softened grain is then passed through a mill to crack or split the corn and partially grind it. The mass is then diluted with water and passed through a long V-shaped tank with a screw conveyer along the bottom and skimming paddles at the top. The germ, or embryo, being now loosened, and since it contains oil is specifically lighter than the bran, gluten, starch, and fibre, rises to the surface and is skimmed off by paddles, while the other solid matters are carried away by the water. The germs are collected, and after again being washed to remove adhering starch, are put through revolving cylindrical driers. The dried germs are ground, steamed, and subjected to hydraulic pressure to extract the oil. The oil obtains a higher price than any of the other ingredients of the corn, and is exported to Europe in bulk for use in soap

manufacture. It is also used in tanning, for paints and for making artificial rubber. The fatty acids derived from it may be saponified with soda ash, which is cheaper to use than caustic soda. A medium-sized factory working 25,000 bushels of corn per day would turn out 100 barrels of oil. This represents 2000 millions of germs used. The residue from the oil-press is considered superior as an oil cake to cotton seed or linseed cake; it contains 20 per cent. of proteins.

The bran, gluten, and starch in suspension are now passed over the shakers, vibrating screens of wire gauze, or coarse mesh silk, the coarser portion being first ground and then returned to be likewise passed on the sieve. The starch passes through the sieve while the bran separated is removed, pressed to squeeze out the water, and sold as press feed or dried to form corn bran containing 14 per cent. of protein.

The starch milk is collected in tanks, whence it is pumped to flow over the settling tables or runs which are about 120 feet long, a foot or two wide, and 6 or 8 inches deep, and with a fall of a few inches only in the whole length. On these tables the starch gradually settles, and forms a compact layer, while the gluten and other residues with some starch pass off to settling tanks. From the latter the water is run off to waste; the sloppy mass of solids, after being pressed and dried, is sold as gluten feed, containing up to 40 per cent. of protein. The starch deposited on the runs, called green starch, which contains about 50 per cent. of water and a half per cent. of protein, is removed with wooden shovels, and may be used for conversion into glucose or is stirred up with water and again passed over the settling runs to secure further purification by sieving and draining. It then contains about 50 per cent. of water, and is dried in stoves at a temperature of 30° to 50° C., until the water content is reduced to 10 per cent. It is ground fine, passed through revolving silk screens, and packed, for use as edible starch and baking powder, for laundry purposes, for giving a stiffening and finish to

cotton and other textiles, for paper, and for adhesive pastes and gums.

Where no caustic soda has been used the starch is described as unchemicalled. This gives a viscous paste, although less so than wheat starch, and is characterized as thick-boiling starch suitable for baking powder and for use as size with textiles. Alkaline treatment has the effect of causing a flocculation of the colloidal gluten and a saponification of the oil so that the starch obtained is whiter and purer. The deposit of this chemicalled starch is diluted in tanks, technically called breakers, to a cream of 22° Bé., and then placed in cloth-lined boxes of 7 to 8 inches cube. When drained to 45 per cent. of water content it is removed and placed on porous bricks to set, then stoved. A brownish crust forms about ¼ inch in thickness which is scraped off. When the water content is reduced to about 30 per cent. it is wrapped in paper and dried for use as domestic laundry starch. This quality is slightly thin boiling; special thin boiling starches are made by treating the wet starch with hydrochloric acid and drying at a temperature not exceeding 65° to 75° C., that is, below the gelatinizing point. By this means some of the starch is converted into dextrine. This quality of starch is in demand for steam laundries and in confectionery works.

**Rice Starch.**—The raw material for making rice starch is the broken rice from the mills in which it is cleaned and polished, and since the removal of the proteins is difficult it is always necessary to use caustic soda. The average analysis of the broken rice as used is: starch 76, other carbohydrates 1·5, proteins 8, fat 0·5, ash 1·5, water 12·5 per cent.

The rice is steeped in caustic soda lye of 0·5 to 1·0 degree Baumé, for 18 hours, and the liquid then drained off. After another like treatment for 12 hours the grains are soft. They are ground between millstones, and the creamy mass of crushed grain is usually passed through a second mill to ensure the absence of lumps that would entail loss of starch. Soda lye is added during the grinding in quantity

such that the resulting cream contains 20 to 28 per cent. of solids. From this cream the starch is separated by sieves or centrifugals. The sieves are rotating drums consisting of a framework covered with the finest silk gauze. These are about 12 feet long, and with a fall of about 6 inches. The axle is hollow and perforated so that jets of water may be played on the inside of the drum to keep the gauze clean. The starch milk passes through into the casing to collect into the settling tanks, while the waste passes out at the projecting end of the drum and is used as cattle food. In the settling tanks the starch is deposited and the protein and fibre remain in suspension and are run off. Centrifugals are to be preferred to settling tanks; they are usually about three feet in diameter, and are run at 1400 revolutions per minute. The basket is not perforated. When a sufficient layer of starch has been formed the surface is scraped and the deposited starch ground up and again centrifugalled. The yield of starch is about 85 to 90 per cent. of that in the rice used.

The recovered starch is mixed with water and weak soda lye and run into a row of wooden boxes, the bottoms of which are perforated and covered with cloth. These boxes are lifted and dropped again to assist the draining. After 24 hours the blocks of compact starch which have formed, and still contain 42 to 44 per cent. of water, are cut into rectangular blocks ready for drying. It is now more usual to employ filter moulds where pressure is used, but it is necessary in this case not to use a milk of more than 1·2 to 1·25 specific gravity, otherwise the finished product has an undesirable rough fracture.

When the above blocks dry a yellow crust is formed about half an inch thick. This is removed at a certain stage and the rest dries white. It has not been found possible to devise means to prevent the formation of this crust. The amount cut away represents about 25 per cent. of the total starch and the whole of this material has to be reworked in the next batch. The starch is generally blued with ultramarine. The blocks remain in the drying chamber

at 30° to 50° C. for two or three weeks. When dry the starch still contains 12 per cent. of moisture, and takes up from the atmosphere another three per cent.

Rice starch has largely replaced wheat starch for many purposes. Owing to the smallness of the granules, it may be used in powdered form with cold water for stiffening linen in laundry work; also for cosmetics. The smallness of the granules led to the selection of rice starch for use in the Lumière process of colour photography. Three portions of starch are dyed, each a different selected colour, and the mixed granules spread over the photographic plate. On viewing the negative by transmitted light objects are seen in their natural colours.

**Potato Starch.**—The manufacture of potato starch reaches its highest development in Germany, where its production far exceeds that of other kinds; a considerable amount is also made in the United States, but our own country has abandoned this source of starch in spite of the manifest advantages to be derived from the association of the industry in small factories attached to farms and worked during the winter months, as is the case to a great extent in Germany, where small factories are the rule, and the waste pulp is utilized on the spot for feeding cattle and the wash, of considerable manurial value, for irrigating the fields. A resuscitation of this industry would tend to reduce the imports of starch at present necessary. The imports of starch, farina, dextrine, and potato flour into the United Kingdom are given below :—

| | 1912. | 1913. | 1914. | 1915. |
|---|---|---|---|---|
| Cwts. | 1,721,977 | 2,135,598 | 1,729,010 | 1,861,207 |
| Value | £1,119,143 | £1,311,044 | £1,069,470 | £1,348,317 |

The average analysis of the potato is: starch 16 to 23, other carbohydrates and fibre 2·5, proteins 2, fat 0·2, ash 1, water 76 per cent. The potato yields more starch per acre than grain, notwithstanding the fact that it only contains about 20 per cent., while wheat contains 60 and rice 83 per cent.; the low percentage of starch is more than

counterbalanced by the great weight of the crop per acre. Thus from grain with a yield of 12·5 cwts. per acre, 8·2 cwts. of starch are produced, potatoes with 125 cwts. to the acre give 22 cwts. of starch, or nearly three times as much. For the decade 1904–1913 the annual production of potatoes in Germany amounted to 50,000,000 tons or 1815 lbs. per head of the population, an average of 5·5 tons per acre, although crops of 9 to 14 tons per acre are recorded. They furnish fresh food, starch, glucose, grape sugar, dextrine, and spirit. In Ireland the crop during the same period was about 5 tons per acre, 591,000 acres under cultivation yielding 3,132,000 tons. The average crop in the United Kingdom averages 5·63 tons per acre.

The value of the potato to the starch manufacturer is dependent on its content of starch, and the value increases in greater proportion than the richness owing to the greater facility in recovering the starch and the smaller loss in that carried away with the diminished quantity of waste pulp. A rough approximation of the value may be obtained by a determination of the specific gravity of the tubers, since this increases with the total dry solids and is more or less proportional to the starch contained. The determination is made by weighing about 10 lbs. of potatoes first in air and then in water and calculating the specific gravity therefrom. Two wire baskets are suspended from one arm of a steelyard or other balance, one immersed in water and the other above. The potatoes are first weighed in the upper basket and then transferred to the lower one and again weighed. More accurate results may be obtained, if necessary, by chemical methods, the starch being hydrolyzed with hydrochloric or lactic acid in an autoclave, and the dextrose produced determined by titration with Fehling's solution. The specific gravity method, however, is usually a sufficient guide, with the assistance of the table.

| Specific gravity | 1·080 | 1·090 | 1·100 | 1·110 | 1·120 | 1·130 | 1·140 |
|---|---|---|---|---|---|---|---|
| Dry solids .. | 19·7 | 21·8 | 24·0 | 26·1 | 28·3 | 30·4 | 32·5 |
| Per cent. Starch | 13·9 | 16·0 | 18·2 | 20·3 | 22·5 | 24·6 | 26·7 |

The potatoes are first passed through a washing machine for thorough cleansing. This is a trough filled with water in which the potatoes are moved along by rotating arms fixed on a horizontal axis, the potatoes rising and falling and rubbing against one another, thus helping to free them from adhering earth. The washed potatoes are now rasped as finely as possible in order to open the cells and release the starch granules contained therein. The rasp or grater is a drum revolving on a horizontal axis at about 1000 revolutions per minute, the periphery of the drum being provided with a number of radially fixed saw blades (Fig. 2), and covered with a casing. The potatoes are torn

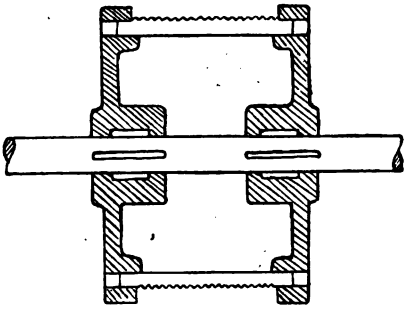

FIG. 2.—Section of Rasping Drum.

by the teeth of the blades into a coarse, frothy, reddish paste, and further comminution is ensured by the mass being compelled to pass between the drum and an adjustable wooden block, and the finer portion is collected in a pit below the machine (Fig. 3). A perforated metal sheet below the drum prevents the passage of coarse particles until the latter are sufficiently reduced in size. Water is added during the operation in quantity equal to the weight of potatoes worked. The rasped mass consists of free starch granules, starch-bearing cells that have escaped disruption, fibre, and dilute juice. The starch is washed out of this material on a sieve by a powerful stream of water, the starch milk passing through, and the fibre and unopened cells that are retained being, in small factories, removed to be used

as cattle food, but in most cases is further comminuted by being ground with millstones and again sieved. The sieves are either flat with an oscillatory motion or formed of a semi-cylindrical trough provided with a series of brushes

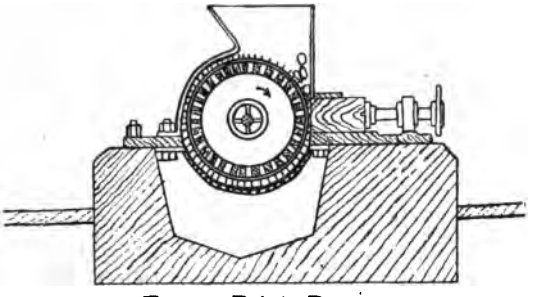

FIG. 3.—Potato Rasper.

arranged radially in a spiral on a horizontal axis (Fig. 4). The starch and juice with the added water pass through the wire gauze to the trough below, and then on to a finer sieve.

The starch milk from the fine sieve is run into settling tanks or through gutters where the specifically heavier starch with any sand present settles out, and the juice and the bulk of the fibre pass away through outlets or by a floating siphon after the starch has become a firm mud. The time required for settlement in the tanks is from 8 to 10 hours. The raw starch thus obtained is yellowish, brownish, reddish, or grey in colour, and so firm that a man may stand upon it to shovel out the mass. It is purified by making it into a cream of 18 degrees Baumé with water and mostly again settled for 6 to 10 hours. It then forms a layer white below with a discoloured scum of inferior starch above. The upper layer is sprayed or scraped away and the operation repeated. The wet starch may be sold as

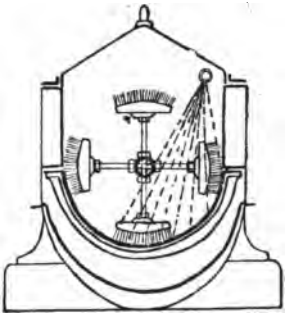

FIG. 4.—Sieve.

such for the manufacture of glucose and dextrin or the moist or "green" starch may be further treated and dried for first quality starch.

The scum is worked to a milk and passed over silk gauze sieves to obtain white starch, or in addition it may be passed along gutters to settle, and thus yield a further quantity of first quality. The water run off into pits forms a deposit which is worked up at the end of the season for inferior grades of starch.

For drying the starch centrifugal machines are used, provided with a filter cloth inside the basket. The starch mud contains 50 per cent. of water, and this is reduced in the centrifugal to 35 per cent. The brownish layer on the surface is scraped off, and the rest is broken up and dried on frames covered with cloth in stoves heated not higher than 35° C., that is, below the gelatinizing point of the starch, until the percentage of water is reduced to 20 per cent. This gives a coarse-grained starch which is the form preferred. It may, when dry, be powdered in mills and sieved to form starch flour, a soft, pure white, glistening powder.

The pulp from the fine washing sieves cannot be profitably worked further for starch. It contains 97 to 98 per cent. of water and contains fibre, insoluble protein, unbroken cells, and potato skin. It is used as cattle food. It is sometimes pressed to reduce the water content to 20 to 30 per cent., and dried to form pulp bran.

The washings, containing nitrogen, potash, and phosphoric acid, are prone to undergo putrid fermentation, and when they cannot be used to irrigate the fields must be purified before they are run into neighbouring water, otherwise disputes may arise owing to disturbance of fishing rights. Besides methods of irrigation for this purification, aluminium hydroxide is used for clarifying them.

In order to prevent the development of bacteria in the starch milk during settling, a weak solution of sulphurous acid is frequently employed, and may be added immediately to the raspings. The use of this reagent is more particularly necessary during warm weather. When diseased, frozen

or unripe potatoes are used the starch is very slow in settling and frequently has an acid reaction. This fault may be overcome by the addition of caustic soda or lime. The latter is safer, as there is less likelihood of an excess of alkalinity when lime water is used. The amount added should be just sufficient to give a blue reaction to litmus.

**Arrowroot Starch.**—In the West Indies the roots of *Maranta arundinacea* are utilized for the preparation of arrowroot. The long, jointed, tap-shaped roots are first soaked in water and washed, then peeled with knives to remove the skin which contains a resin that would communicate an unpleasant taste to the starch, and also discolour it. The roots are then pulped by crushing them, and the pulp is washed through perforations in a cylinder with water to separate the fibre. The milk is filtered through muslin, settled and washed repeatedly, and finally dried in shallow copper pans covered with muslin to keep out dust. The roots yield about 19 per cent. of starch and give 22 cwts. of air-dried starch, containing 14 per cent. of moisture, per acre.

**Sago.**—Sago is extracted from the pithy interior of *Sagus Rumphii* and other palms. The trunks are cut into pieces about two feet long, which are split in half and the pith taken out and kneaded in water. The starch is then washed, and the moist sago formed into pearl sago by forcing it through sieves or rolling in a cloth, and then heating in pans with a little vegetable fat. A single tree yields six or seven cwts. of sago. It is chiefly exported from Singapore. Artificial sago is made from potato starch by pressing the moist starch through coarse sieves, dusting the pellets with dry starch and heating until partly gelatinized.

**Tapioca.**—The tubers, about 30 pounds in weight, of *Manihot utilissima* or *Cassava* in Brazil are washed, peeled, and pounded to pulp and pressed in baskets to wash out the starch. This is then heated on iron plates at a gentle heat to volatilize the hydrocyanic acid. The grains swell, burst, and agglutinate.

**Soluble Starch.**—When starch is allowed to remain

for twenty-four hours in contact with 12 per cent. hydrochloric acid and freed from acid by washing, it dissolves in hot water without forming a viscid paste. The solution deposits the soluble starch on cooling as a pasty mass. It has no action on Fehling's solution. Many reagents have been employed at various temperatures to produce soluble starch. Most of these preparations, however, contain dextrose, dextrin, or maltose, as well as modified starch, and possibly starch esters. Starch is heated with 1 per cent. formic or acetic acid at 115° C. for 5 to 6 hours, the excess of acid distils off so that it is not necessary to neutralize. Starch, when heated with caustic soda solution, becomes pasty, and when the alkali is neutralized with sulphuric acid the paste is miscible with water. Chlorine gas, bleaching powder, persulphates, and ozone have all been proposed for rendering starch soluble.

**Uses of Starch.**—Besides the use of starch and materials containing starch for food, and the production of beer and alcohol, and for the manufacture of glucose, dextrin, and caramel, it is used in laundry work for stiffening linen. The starch is gelatinized, and the hot iron converts the starch into a shining layer of dextrine. Potato starch, the cheapest form, when mixed with borax forms a starch glaze. It is also used mixed with powdered stearic acid or paraffin. Rice starch may be used mixed with cold water. Wheat starch is used as a paste for bill-posting, paper-hanging, and bookbinding, owing to its superior adhesive power. Starch is used for applying mordants in calico printing, and starch and rosin for sizing paper. Thin starch paste is used for finishing and weighting calico and cloths. Rice starch is used as a face powder, and for dusting moulds in the metal foundry, and for moulds in making fondants in confectionery. Starch is used in chemical titrations to indicate the presence of free iodine. For this purpose it is preferable to use a form of starch soluble in cold water. This may be made by running starch paste in a fine stream into absolute alcohol or acetone. The precipitate is dried at a moderate temperature.

## Section II.—DEXTRIN

Commercial dextrin, or British gum, is usually met with as a yellow or brownish powder which has a mealy odour slightly reminiscent of newly baked bread but at the same time somewhat repulsive. It is also produced in the granulated form as brittle, semi-transparent lumps resembling gum arabic, for which dextrine forms a cheap substitute. This is sometimes described as "crystallized" gum. Occasionally it is sold as a fluid dextrin, an opaque, milky syrup. The different qualities are made to suit the various purposes for which it is used. It dissolves in cold water, forming a viscous solution with adhesive properties, and is used for gumming labels, envelopes, and postage stamps, for finishing cloth, and as a thickening for mordants in calico printing. The finer qualities are also used in confectionery and in various foods.

Dextrins are manufactured by heating starch, either alone or after treatment with acid, until the desired degree of dextrinification is reached. The preliminary change is indicated by the reaction with iodine, but for the control of the more highly dextrinized products the only methods yet available are the feel, colour, and appearance when moistened on a glass plate. It is consequently extremely difficult to imitate exactly a given sample of dextrin, and for the production of the few constant qualities made by a given firm reliance is placed on the control of preliminary drying, the percentage of acid used, and regulation of the temperature and rate of heating.

**The Roasting or Torrefaction Process.**—The previously dried starch is heated in trays in a stove to a

temperature of 180° to 250° C., until the transformation is complete, or the starch is fed into rotating iron drums heated by oil or by direct fire and passes out as dextrine, the process being thus continuous. The higher temperatures give a darker product, but this is more completely soluble than the paler qualities. A better product is obtained by heating the starch in a closed vessel provided with stirring gear and heated below with superheated steam. After roasting, the product is ground or crushed by passing between rollers and then sifted.

**The Acid Process.**—Starch is treated with 0·2 to 0·25 per cent. of hydrochloric or nitric acid and then dried and powdered to secure uniform acidification, after which it is passed to the heating apparatus, consisting of covered pans or drums heated by oil, steam, or direct fire to a temperature of 100° to 150° C. In these it is stirred during the heating until the change is completed, which may require from 1 to 2 hours. The dextrin, after roasting, contains about 3 per cent. of moisture; this is raised to the usual 10 or 12 per cent. present in commercial samples, by exposing it to a moist atmosphere. It is then sifted and well mixed. The colour, flavour, and odour are said to be improved by passing ozonized air over the starch during the heating.

So-called "crystal" gum is made by decolorizing a hot solution of slightly dark dextrin with animal charcoal and evaporating either to dryness, and grinding the flakes obtained to powder, or to such a density that the solution on cooling forms a pale transparent solid mass.

Substances which are applicable for many purposes where dextrin is used may be formed by esterifying starch with acetic acid.

According to Traquair acetic acid in solution has little action on starch, but when an excess of glacial acetic acid is heated with starch, dried to a content of only 3 or 4 per cent. of moisture, a product is obtained insoluble in cold water, so that the excess of acid can be removed by washing by decantation. To prevent formation of products soluble

in cold water (dextrins), limited heating, 8 hours at 90° C., or 2 to 3 hours at 120° C., is necessary, thus allowing the excess of acid to be removed by washing. When swelled by boiling with 5 or 6 times its weight of water, feculose, as the acetylated starch is called, gives a clear solution which is permanent and does not set or revert on standing for a few hours. A portion of the solution poured on a glass plate dries to a clear, continuous film equal to that obtained from the finest gelatin, and, in fact, technically, feculose may be described as non-nitrogenous gelatin.

**The Uses of Dextrin.**—In addition to the commercial uses of dextrin already mentioned, it is largely employed as an adhesive in paperhanging and bookbinding. "Gloy" is said to be a mixture of dextrin with magnesium chloride. When it is employed as a thickening agent for colours and mordants in calico printing, it is usually mixed with starch. Shoemakers' paste is also a mixture of dextrin with starch. The glaze produced on linen, and the gloss on the crust of bread, are due to the dextrinization of the starch by heat. The corn syrups used in making confectionery, preserves, and sweetmeats are preferred to sugar or cane syrup owing to the absence of grain.

## Section III.—GLUCOSE

In 1803 Proust established the fact that a sugary substance, grape sugar, could be extracted from the juice of the grape. In the following year Bouillon Lagrange observed the change which starch undergoes when submitted to high temperatures, and used the resulting product as a substitute for gum arabic for fixing dyes and for making inks. In 1811 Vaucquelin incidentally described the formation of dextrin when starch was torrefied, gave an account of its properties, and pointed out the commercial possibilities of the substance. Kirchhoff, the same year, requiring gum arabic for some experiments he was making on the manufacture of porcelain, and being unable to procure it, made use of the substitute prepared from starch. Then, probably with the intention of obtaining clearer and less highly coloured solutions, he tried boiling the starch with dilute sulphuric acid, and found that the solution contained sugar instead of the adhesive gum which he required. Braconnot found that the same sugar was formed by treating linen rags in a similar manner. At the time of Kirchhoff's discovery colonial or cane sugar was almost unobtainable on the continent except at fabulous prices, owing to the embargo then existing against the entrance of all products from England and its colonies, and the method was put to practical use almost immediately after its first publication.

Kirchhoff's method as described by Nasse was to use 100 parts of starch, 400 parts of water, and 1 part of sulphuric acid. It was recommended to dilute the acid with half the above quantity of water and bring to a boil. The starch was made into a milk with the rest of the water and poured gradually into the boiling acid mixture which

was kept stirred and at boiling temperature for 36 hours in succession in open vessels, the water lost by evaporation being renewed. Neutralization with calcium carbonate, followed by filtration over bone-black and evaporation of the sugary liquid to a syrupy consistency, completed the operations. The syrup left for a certain time began to crystallize, usually about the third day, and was separated from the mother liquor through filter bags and pressing. From 100 parts of starch Kirchhoff obtained 90 parts of solid sugar or 120 parts of very sweet syrup. The sugar, evidently supposed to be sucrose, is described as less sweet than cane sugar, with a peculiar after-taste which, however, can be removed by refining. The diminished sweetness was accounted for by the higher percentage of water of crystallization which could not be removed at higher temperatures. The sugar resembled more that obtained from grapes, but was considered superior to the latter. The sulphuric acid was found to act on starch in this way in all proportions, provided it was properly diluted, and it was also observed that at first a gummy substance was formed, and sugar was only obtained on continuing the boiling. Vogel, of Paris, repeated the experiments in 1812 and concluded that sugar and gum are formed in variable proportions, the gummy matter being precipitable from the syrup by 85 per cent. alcohol; that heat alone is not sufficient to transform starch into sugar, and that the sugar formed is a true sugar since it is capable of undergoing alcoholic fermentation; also that the syrup when properly purified contains no sulphuric acid. The identity of starch sugar with grape sugar was established by Saussure in 1814; and by combustion he also found that starch differed from sugar only by the elements of water.

When the continental blockade could no longer be maintained against the gigantic system of smuggling to which it had given rise and was removed in 1814, the colonial products flooded the market and brought down the price of sugar to extremely low levels, especially as this

was aided by competition of beet sugar, the manufacture of which had now considerably advanced, and moreover the production of starch sugar was still beset with difficulties so that this manufacture was practically abandoned. It was only with the gradual introduction of the use of glucose in the fermentation industries through the labours of Lampadius in Germany and Dubrunfaut and Payen in France that the manufacture took on a new development.

Starch sugar finds no application which common crystallized sugar could not fulfil, and it must always be considered as merely a substitute; it is, however, directly fermentible, while cane sugar is not. Its sweetness is only two-thirds that of cane sugar; in nutritive value the two are equal.

**Manufacture.**—In the manufacture of glucose syrup and starch sugar by means of sulphuric acid the following operations are necessary: the conversion or saccharification, which consists in boiling the starch with dilute sulphuric acid, generally under pressure; removal of the acid by neutralization and elimination of the sulphate of lime produced from it; concentration of the filtered liquor and refining and concentration to syrup with subsequent separation of the crystallized sugar.

In the earlier processes the boiling was conducted in strong wooden vats, either open or constructed, so that they could be heated under pressure, and provided with a steam coil. The required quantity of water with the previously diluted sulphuric acid is placed in the vat and heated to boiling. The starch milk is then run in gradually at such a rate that boiling does not cease. The starch milk is prepared in a separate stirring vat; since starch deposits quickly from the starch milk it must be kept constantly stirred. It is necessary to avoid formation of a paste while the starch milk is run into the boiling acidulated water. For each 2 cwt. of air-dry starch 40 to 50 gallons of water and about 4½ lbs. of sulphuric acid are required if syrup is to be produced and about double the amount of acid if solid sugar is to be made. Half of

the water is used to make the starch milk. Where the "green" or wet starch is used a larger quantity is taken to allow for the moisture present.

When the whole of the starch-milk is in, the boiling is continued until transformation is attained. A shorter duration of boiling is needed for the production of syrup than for solid sugar. During the boiling a very disagreeable and penetrating odour is developed, especially when potato starch is used.

During the boiling the liquid is tested with solution of iodine, and afterwards with alcohol. When a few drops of the liquid in cold water no longer give a violet or reddish colour with iodine the conversion of the starch into dextrin and glucose is finished. The alcohol test is now applied by adding one or two volumes of alcohol to one of the liquor, and from the quantity of precipitate formed the amount of dextrine still unconverted may be judged. A convenient and ready means for determining the complete conversion into sugar is still a desideratum. The liquid is now neutralized by the addition of chalk; lime cannot be used as it would destroy the glucose. When litmus paper shows a perceptible decrease of acidity the liquid is boiled for a short time before more chalk is added. The liquid when neutral is allowed to deposit the sulphate of lime, and the liquor is then drawn off from the depositing vats, otherwise it is passed through a filter press. The clear liquor is evaporated either over a direct fire or by steam until it reaches a concentration of 30° Baumé, or until a separation of sulphate of lime renders it necessary, when it is either allowed to settle or is passed through a filter press. It is then evaporated to a dense syrup of 40° to 45° Baumé in vacuum pans. In order to improve the colour and flavour it may be filtered through animal charcoal before the final concentration; this treatment is indispensable for producing a solid, white sugar. If the conversion of the starch into sugar has been very complete the resulting syrup will soon begin to crystallize.

The manufacture of glucose from maize in the United

States has attained enormous proportions, rendered possible by technical improvements in the process of manufacture and the growing demand for the various products of the industry. The starch is heated with hydrochloric acid under pressure in the converters, strong vessels of hammered copper, which may be 20 feet high and 6 feet in diameter. Water and the acid are introduced and raised to the conversion temperature, and the starch suspended in cold water is pumped into the boiling acid. As conversion goes on during the pumping there is no formation of clots owing to accumulation of starch paste. The extent to which conversion is carried depends on the purpose for which the product is designed, and is regulated by the temperature and duration of the conversion. For grape sugar, the solid crystalline form of glucose, the conversion is carried to completion; for liquid glucose or syrup, the change is not allowed to proceed so far, and a certain percentage of dextrin is left unconverted. The products are glucose (dextrose), maltose, and dextrin. It takes 12 to 15 minutes to pump a charge of 1000 gallons into the converter, and a little longer to complete at a maximum temperature of 132° C. As soon as a sample shows a satisfactory test the operation is stopped by releasing the pressure, which at once causes a fall in temperature. The product is run into vats, and neutralized with soda ash. The small quantity of hydrochloric acid present is thereby converted into common salt which has no effect on the taste of the glucose in these proportions. The neutralized liquor is passed through filter presses to take out fibre, protein matters, etc., and then through evaporators in which it is concentrated to a density of 30° Baumé. It is then run in succession through three filters filled with animal charcoal, each cistern being 10 feet in diameter and 24 feet deep, and containing about 35 tons of char, the total depth traversed is thus 72 feet. The proportion of char is more than is used in refining cane or beet sugar, and the liquor issues practically colourless and brilliant. When the desired quantity of liquor has passed through the char, and its

decolorizing power begins to fail, the sweet is washed off and the char revivified during its passage by gravity through the pipes of a kiln heated by oil or coke. The revivified char is sieved to remove dust and the loss made up by the addition of new char. The filtered liquor is now concentrated in a vacuum pan or Lillie apparatus until it reaches a density of 42° to 45° Baumé. It then forms a clear, viscous syrup. In this form the less highly converted product, containing about 40 per cent. of dextrine, is sold as glucose or corn syrup, for use in confectionery and for table syrups. The more completely saccharified liquors, after concentration, crystallize on cooling to a tough, solid mass of white or yellow sugar called solid glucose or grape sugar. This is ground up or chopped into fragments and used in brewing as an adjunct to malt, and for the manufacture of caramel. It is also employed for petiotizing wines that are deficient in sugar. Pure anhydrous grape sugar is obtained in crystalline form by the process introduced by Behr. Into the warm glucose syrup a small quantity of anhydrous glucose is quickly and thoroughly incorporated to act as seed to induce crystallization, and the mass allowed to stand in crystallizing troughs. After the crystals are formed the whole is stirred up to a magma and the mother syrup separated by centrifugal treatment. The dried crystals contain less than 1 per cent. of ash and of dextrin and 4 per cent. of water.

A process for making glucose which possesses special features is one due to S. H. Johnson. Whole, crushed, bruised, or broken grain or other granular amylaceous material is used in a condition such that steam may be able to permeate a mass of it. Rice or other grain in the form of flour is unsuitable, as the gelatinous paste which would be formed by it would be impermeable to steam. The grain is first macerated in 2 per cent. hydrochloric acid, which dissolves proteids and carries off essential oil. The acid is drawn off below and water then percolated until neutral to litmus, when the mass is drained and again macerated with 1 or 2 per cent. hydrochloric or other acid until the grain has

taken up enough acid to effect conversion into glucose, the proportion varying with the nature of the grain. Rice usually requires about 0·25 per cent. of actual HCl. The drained material is then introduced into the converter, a strong vessel of wrought iron lined with lead or with a silicate enamel capable of resisting the action of the acid, and provided near the bottom with a perforated diaphragm. The prepared grain introduced into the converter retains its form, and being porous is readily permeated by the steam entering below the diaphragm, so that the whole mass is raised to the conversion temperature before the porosity of the grain is destroyed by the water condensed from the steam, without the necessity of any stirring arrangement. The contents of the vessel become liquid, and at pressures of 50 to 100 lbs. rice is converted in from 5 to 15 minutes as indicated by the iodine test on samples withdrawn during the operation. The liquid is then run off into coolers, stirred to assist the cooling, and neutralized with dry sodium carbonate. The mass will become more or less solid after standing, provided the material did not contain an undue amount of water when introduced. The glucose thus prepared is very pure and free from any bitter or empyreumatic flavour, and is used for brewing and distilling. Sago and tapioca which have already undergone purification during their manufacture may be used without the preliminary treatment, and are much used for making glucose.

The Board of Trade bulletin gives the value of starch of all kinds exported from Germany in 1912 at £555,200; from Austria-Hungary (1913), £46,010; and from the United Kingdom (1913), £88,200. The value of the exports to the principal colonial and neutral markets was: Germany (1912), £288,350; Austria-Hungary (1913), £27,580; United Kingdom (1913), £72,610. Of the total German exports, potato starch, green fecula (wet starch), and dry potato starch powder (potato meal) accounted for £220,350; dextrine, roasted starch, adhesive and surface dressing substances containing starch, gluten and gluten powder, £187,100; rice starch, £117,750; and starches of maize,

wheat, etc., £30,000. More than half of the total Austrian exports consisted of paste, size, and similar starch-containing sticking and dressing substances, and rice starch and rice starch meal.

The corn (maize) manufactured into corn products in the United States amounts to 50 million bushels per annum. From this are produced 400,000 tons of glucose, 300,000 tons of starch, 115,000 tons of grape sugar, 300,000 tons of gluten feed, 37,000 tons of oil, and 45,000 tons of oil cake; 300,000 bushels of potatoes are used in the starch factories.

**Chemical Synthesis of Sugars.**—The manner in which carbohydrates are formed by the vital activity of plants from carbon dioxide and water offers a difficult problem for synthetic chemistry. Moreover, it is not the simpler carbohydrates which appear, but starch is the first visible product, and sucrose the first sugar which can be detected. The first step towards a solution was afforded by the observation that on treating formaldehyde with lime water a sweet syrup was obtained, having the properties of a sugar due to the aldol condensation, whereby the carbon atoms of separate molecules of the aldehyde link together in a chain. In a similar manner glyceraldehyde and dioxyacetone gave a syrup containing α-acrose, which was shown to be inactive fructose. This was converted into the corresponding racemic mannonic acid, and the dextro and lævo forms separated by fractional crystallization of the alkaloid salts. From dextro-mannonic acid both *d*-fructose (ordinary levulose) and *d*-glucose were prepared. There is considerable evidence in favour of the view that carbohydrates as formed in the plant may originate from formaldehyde through the catalytic decomposition of carbon dioxide by chlorophyll under the influence of sunlight, the formaldehyde being at once polymerized into carbohydrates by the protoplasm of the cells.

## Section IV.—MALTOSE

The main product of the action of malt diastase on starch paste is maltose, which was first observed in 1819 by De Saussure, who isolated the unknown sugar from the hydration products of starch and described its crystalline character. It was further examined in 1849 by Dubrunfaut, who gave it the name "maltose." He found its opticity was three times that of glucose, and that it was the sugar first formed when acids act upon starch. No further notice was taken of these facts until C. O'Sullivan in 1872 rediscovered the sugar and brought the matter into prominence. The process of the saccharification of starch by acids had by this time become firmly established, and no industrial application of the use of diastase for the preparation of the sugar or syrup appears to have been made until Cuisinier in association with Dubrunfaut in 1883 patented a process for preparing maltose either as syrup or crystallized. His procedure was to liquefy starch at a temperature of 70° to 80° C. with 5 to 10 per cent. of an infusion of green malt, and then to carry out the saccharification at 50° C. with from 5 to 20 per cent. of the diastase, after making the solution faintly acid with hydrochloric acid. The solution was filtered through paper supported on linen cloth. After standing from 12 to 15 hours it was concentrated to 28° Baumé, filtered and further concentrated to 38° Baumé, filtered through animal charcoal and allowed to crystallize. A sample of syrup made by the Maltose Company of Brussels was found on analysis by Märcker to contain, water 19·8, maltose 78·7, non-sugar 1·5, and no dextrin. The process, however, did not prove a commercial success. More recently the subject has been investigated by Duryea, who

attributes the failure to the use of crude raw materials, ground maize, or impure starch flour, and in such a state that, owing to the thick boiling condition and consequent formation of viscous paste, concentrations equal to those of the light liquors of the glucose industry could not be realized, and the quantity of malt required was excessive. The presence of a large percentage of undesirable nitrogenous and other extraneous matters increased the cost of refining. Duryea uses a thin boiling starch made to 16° Baumé, neutralized, boiled, cooled to 59° C., and saccharified with 5 per cent. of malt, calculated on the dry highly modified starch. He finds that the product has a more luscious taste when made into hard candy than the similar sweets made with glucose, and maltose candies have less tendency to attract moisture and run. High-grade industrial maltose syrups produce an impression of superiority upon the palate as compared with glucose syrup, which is never used alone, but only for mixing with cane or sorghum syrups to make table syrup.

The intention of Cuisinier was that the maltose products should be used for brewing purposes, for making liqueurs, sweetening wines, and in the distillery. It may be noted that the proportion of maltose in commercial glucose generally exceeds that of glucose (dextrose), the content of the latter may be as low as 12 per cent.

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# PART II.—SUGAR

## SECTION I.—CANE SUGAR

THE universal sweetening substance in use at the present time is cane sugar, whether derived from the sugar cane, which formerly furnished the whole supply of sugar, the beetroot, sugar maple, sorghum or sugar palm, for the purified product from any of these sources is the chemical compound sucrose, $C_{12}H_{22}O_{11}$. The oldest sweetening material used by mankind, however, either for flavouring food or as a medicament, was without doubt the honey of wild bees. The sugar cane became known at a much later period, and at first it was merely chewed or eaten for the sake of the sweet juice; there was no actual preparation of the sugar contained in it.

The original home of the sugar cane was probably either the East Indies or the Pacific Islands, and the art of extracting sugar from it was certainly discovered in India, since the word "sugar" is common to all languages, and is derived from the Sanscrit "sharkara" (Pracrit sakkara), which meant a pebble, and was used to denote sugar. The first Europeans to become acquainted with the cane were the followers of Alexander the Great on his expedition to India B.C. 327, for the early writers in describing this undertaking speak of a honey which did not come from bees. Later it was known to the Greeks and Romans as Indian salt. The sugar cane was already known in China in 200 B.C., but it did not come into general use until after the Persians had become acquainted with it in the fifth century. Persia was long the principal seat of the sugar industry, and, where now on its arid plains only the ruins of old sugar mills and sluices

remain, there were formerly extensive cane fields. It was used chiefly for medicinal purposes, and the first successful attempts at refining it were made at Gondisapur. The Arabs who conquered Persia in 640 developed there considerably the culture of the cane and the industry of sugar, and then transplanted it to Arabia and the countries successively conquered by them—Egypt, Morocco, Sicily, and Spain. From the chemical knowledge of the Egyptians the manufacture of sugar received important improvements. They had known for centuries the manipulations and apparatus for filtration, distillation, crystallization, etc. They knew how to clarify plant juices by the addition of albumin, boiling and filtration, or by treatment with alum; how to precipitate albumin by tannin and the use of hydrometers. As they were aware of alkaline working, their sugar bore transport by water, and for centuries was a highly esteemed commodity. The Egyptians were the real founders and inventors of refining. Even the Chinese were only able to prepare sugar as a pasty mass by drying cane juice in the sun, and first learnt the manufacture about 650, and refining from their conquerors, the Mongols, in the thirteenth century. The Arabs having carried the cultivation of the cane westwards along the shores of the Mediterranean, from 1095 the Crusaders made sugar widely known and extended its use everywhere. The Venetians trading with the Arabs were actively engaged in the supply of sugar to Central Europe. A sudden change occurred in the sugar industry with the capture of Constantinople by the Turks in 1453, and their subsequent conquests in the Black Sea, the Ægean, and Egypt closed access to Asia for the Venetians; wherever they entered as conquerors the Turks arrived as devastators of the conquered countries and of all ancient civilization. The sugar cane disappeared from all these parts or became of no importance. Its cultivation has not reappeared in any of these countries except in Egypt, where Mehemet Ali, in 1820, reintroduced it. Meanwhile the Portuguese had brought cane from Cyprus to Madeira and the other islands off the coast of Africa, and the sugar

industry flourished there, aided by cheap labour due to negro slaves. This sugar by its cheapness competed with the Mediterranean for 300 years in provisioning Europe. They also discovered round the Cape the route to India, and the costly route through Asia being no longer necessary, the great market for sugar was transferred to Lisbon, so that in 1515 Venice had to buy its sugar there. During this time America had entered the producing countries for sugar. Columbus, in 1493, on his second voyage took cane from the Canary Islands to San Domingo; thence it spread to Cuba, Mexico, and Peru. By 1553 Mexico exported sugar to Spain. In 1532 the sugar cane was taken to Brazil from Madeira. In 1600 there were 120 sugar factories in Brazil, and its production ruined that of Sicily and threatened that of Madeira to such a point that Portugal had to establish an import duty to protect its old colony. Important Dutch houses founded branches at Lisbon to import direct their colonial commodities. Sugar was sent from Lisbon to Antwerp and thence up the Rhine; numerous refineries were established at Antwerp, but the war of independence of the Low Countries (1566–1609) decided many Dutch to leave, and the industry was transferred to Amsterdam, where sugar refining prospered greatly. In 1580 Philip II. of Spain, having taken possession of Portugal, endeavoured to prevent the Dutch, with whom he was at war, from commerce with Lisbon; the result was different from what he sought. The Dutch colonies entered into direct relations with the Indies and Java, founded a company, and soon conquered all the Asiatic colonies of Portugal. They also attacked Brazil, devastated the plantations, and soon began to make sugar themselves in the conquered country. When Brazil was restored to Portugal by treaty (La Hague, 1661), the Dutch still remained. On their expulsion they went to the West India islands, where the manufacture was so bad that it did not pay to send the sugar to Europe; they improved it and became prosperous. In 1791 the revolt of the slaves of San Domingo stopped all industry there, but was of benefit to Brazil, Guiana, Jamaica, and Cuba; Louisiana also

profited, although its production was backward on account of the unfavourable climate. In Mexico and Cuba the Spanish maladministration had at the end of the sixteenth century completely ruined the sugar industry. The manufacture was introduced into the Far East, principally Manilla, towards 1750; in 1800 Manilla already exported sugar. In 1319 this country obtained about fifty tons of sugar from Venice, and sugar refining was introduced in the sixteenth century, for in 1544 there were two refineries in London.

British India is estimated to produce about 2,600,000 tons annually of cane sugar and gur (crude sugar), and nearly 500,000 tons of palm sugar, and consumes over 3,750,000 tons, so that the deficit has to be imported, chiefly from Mauritius, Java, and Europe. About 25 per cent. is lost in the canes through bad cultivation, and another 25 per cent. in manufacture through overheating and in the megass burnt. Efforts are, however, being made by the Agricultural Departments to improve the cultivation and the processes of extracting cane juice and of sugar manufacture. Java produces an average of 4·3 tons of sugar per acre, and at this rate India could produce 10 millions of tons of white sugar per annum. Java is an example of the successful application of energy and intelligence aided by science and organization. They not only make the finest raw sugar, but a considerable amount of white sugar, the total production amounting to 1,300,000 tons. The yield per acre is still surpassed by Hawaii, where 5·6 tons of sugar is reached, and the total output approaches 500,000 tons. Cuba produces about 2,500,000 tons, some of its huge factories dealing with 300,000 tons of cane per annum. The total cane sugar crop of the world for 1913–1914 is estimated by Willett and Gray at 9,773,348 tons.

*The Plant.*—The sugar cane, *Saccharum officinarum*, which belongs to the natural order Gramineæ, is a gigantic grass reaching about 15 or 16 feet in height, and grows in all tropical and sub-tropical countries. It is most favoured by a hot moist climate with a dry ripening season, and any

lowering of the temperature is prejudicial to its growth. The plant consists of root, stem, leaves, and flowering shoot called the arrow. As in all grasses the roots are fibrous and wide-spreading, never branching or growing to any great size, although they extend to distances up to three feet, dependent on the condition of the soil. The larger roots fix the plant in the soil and act as channels for the transference of water and food absorbed by the root hairs. These fine rootlets take up food in solution only and possess a selective power so that only suitable matters are absorbed for the nourishment of the plant. The root stock is made up of a number of underground shoots giving rise by tillering to numerous stems and producing the tufted habit and forming the stools characteristic of the sugar cane as of other grasses. The stalk consists of joints averaging about six inches in length, and separated by the knotty parts or nodes. The node extends transversally right through the cane, forming a tangled felt of fibres; the internodes are composed of pith, soft parenchymatous tissue, the cells of which are filled with the sugar juice, and traversed by numerous fibres, the fibro-vascular bundles. The rind of the stem is hard and polished, contains silica, and is impervious to water. Just below each node is a narrow, whitish band formed of white powder, really rods of wax. Above this is the scar of the fallen leaf, and next the bud, or eye, and again another band encircling the stem with rows of semi-transparent dots indicating the presence of sleeping roots. The latter arise from within the stem, and when the cane is exposed to excessive moisture, or the node is planted in moist earth, they force their way through and form the roots. The buds are arranged under the base of each leaf on alternate sides of the stem, and are capable of growing into a stem of the future cane plant. The leaves, alternate as in other grasses, consist of a sheath encircling the stem, and the blade often coloured along the midrib. The leaf sheath is often provided with stinging hairs. The fibro-vascular bundles of the stem are the channels through which water and food pass from the roots to the leaves, there to be elaborated, under the action of

sunlight, by combination with carbonic acid absorbed from the air, into sugar and albuminoids, and then pass down into the stem where the sugar is stored in solution in the juice of the cells. The leaf is thus the workshop of the plant, where the materials for its life-functions are elaborated, and by the transpiration from those cells in contact with the air a considerable evaporation of water occurs, determining the upward current of water and dissolved food materials from the roots. At the time of maturity the elongation of the stem bears at its apex a sheath, from which bursts forth the arrow or inflorescence, a grey or white cluster composed of countless minute silky flowers. The ovary, after fertilization, becomes the fruit or seed. Previous to 1887 the general opinion prevailed that the sugar cane had lost the power of producing fertile seed through the long-continued practice of propagating by cuttings, but about that time Harrison and Bovell in the West Indies, and Soltwedel, in Java, found that seedling canes could be raised. A number of such canes are now being experimented with, the object in view being to rear varieties capable of yielding larger crops of sugar and of greater resistance to the attacks of disease. In order to secure this end it is necessary to avoid chance pollination, so that the source of the pollen must be controlled as well as the seed-bearing cane, and this presents some difficulty, owing to the extremely minute size of the flowers and the fact that they are borne on the summit of a slender stalk some fifteen feet high.

For planting cane it is mostly the tops or upper joints of the cane, after removal of the head, that are used, but frequently the whole cane is cut into short lengths of two or three joints each for this purpose. These pieces are planted in furrows, or in holes, in rows several feet apart at intervals of one or two feet. The land requires to be well drained, although a considerable supply of water is required for the growth of the cane, and where rain is deficient irrigation is necessary. When the canes are from one to three months old they are manured; they are moulded up as they grow to keep the root under the surface, and the

necessary weeding and tillage is done. At six to seven months the cane stool is sufficiently developed to be left without further operations except that, as maturity approaches, the dead leaves or trash are removed. The cane being thus more open to air and sunshine maturity is hastened, and the dead leaves help to retain the moisture in the soil and prevent the growth of weeds. The usual period to reach maturity is sixteen months for plant canes and twelve months for ratoons; but in countries with a cold season the period is reduced to eight or nine months, as in Louisiana. When the cane is cut and the stool is allowed to remain, shoots arise from it and form a new growth called ratoon canes. This may be repeated for several years in succession on suitable soil. In Java the Government allows only the growing of plant canes, and these are alternated with crops of rice and beans for native use. The most suitable soil for the cane is clay and loam, which allows of suitable aeration and drainage, yet retains sufficient moisture for the plant growth. The mineral constituents removed from the soil by the cane are small in amount and less than 0·5 per cent. Lime is important, and the principal fertilizers necessary are those containing potash, phosphoric acid, and nitrogen. Since the residual fibre or bagasse is used for fuel, and therefore lost to the soil, as much of the trash as possible is returned to it, and the humus or vegetable debris is supplemented by the growth of catch crops between cutting and replanting. These are usually leguminous plants, and by ploughing in the fully grown plants, green-soiling as it is called, the nitrogen content of the soil is also increased. The time of harvest of the cane crop varies for the different tropical regions, and thus the sugar markets of the world receive a succession of supplies as the various crops mature. The average composition of the cane varies from 7 to 16 per cent. of sucrose; reducing sugars up to 1·5, fibre 10 to 15, ash 0·4 to 2, water 69 to 75 per cent., and the yield being from twenty to forty tons per acre, the sugar obtained may amount to about four tons per acre or more than double what is recovered from the growth of beetroot.

**Varieties.**—There is a large number of varieties of canes under cultivation chosen for their special suitability for a particular soil or climate, or for their high content of juice. The names of these various kinds of cane, however, show great confusion, since different names have been applied to what is the same variety; but grown in localities wide apart and the lack of communication between these regions has led to the perpetuation of the name given. The distinctive characters of the varieties are the colour of the stem and leaves of the mature cane, the size, form, and position of the buds, and the length of the internodes, with other differences. Of the white, yellow, and greenish canes the more noted are the Otaheite, Louzier, Salangore, and Bamboo; the White Transparent, White Java, and Rose Bamboo. The striped canes include the Striped Tanna, Otaheite Ribbon, Cheribon, and Java Yellow. Of the dark-coloured canes there are the Black Tanna, the Louisiana Purple, and Java Cheribon.

**Seedlings.**—The object in view in growing seedlings is to get varieties with new characters, and then continue the selection by cuttings until they are found stable and fit for industrial cultivation. In reproduction from seed the plants from the same mother show greater variability, while those from cuttings are remarkably fixed in hereditary characters. It takes about five years to settle the value of a seedling variety. The first plants from seed are small and seem inferior, they are likewise poor in sugar, but the plant canes grown from these soon begin to reach a normal character, and it is here that real selection is made, based on the form, height, and colour of the stem, the length of the internodes, the content of sugar, quantity and purity of the juice, and resistance to the attacks of disease and capability of furnishing successive ratoon crops.

**Pests and Diseases.**—When the cane plant is weakened by drought or by defective soil conditions it becomes more susceptible to the attacks of fungi and microbes, particularly if the hard outer rind is broken or injured, as by excessive trashing or when gnawed by rats, and a great deal of the time and attention of the experiment stations is devoted to

the study of the diseases to which cane is subject, and to devising means to prevent or counteract the damage. The ends of cuttings used for planting are particularly open to infection, and when these are sent from distant parts they are first soaked for 24 hours in Bordeaux solution, dried, and then packed in animal charcoal with a little copper carbonate; this enables them to keep sound for a long period.

The moth borer lays its eggs on the leaf, and the caterpillars make their way through the soft parts of the rind near the joint or at the eye, or the terminal point and tunnel along the stalk. Fungus pests then enter and spread. The beetle known as the shot borer makes small holes in the rind of weak or diseased canes. The rind fungus causes dark discolorations on the stem. The root fungus, *Marasmius sacchari*, produces dark red spots on the root and lower parts of the stem, and the canes gradually die. The sereh disease in Java has caused enormous damage. It is marked by a general dwarfing of the growth and a reddening of the stalk and tissues. Although hereditary, and apparently not infectious, the disease appears to be due to special soil and cultural conditions rather than to micro-organisms. In the gumming disease drops of a gummy substance exude from the fibro-vascular bundles when the cane is cut across; growth ceases, and the leaves turn yellow. It has been shown to be due to the bacillus *Pseudomonas vascularum* against which certain varieties of cane are immune. The pine-apple disease is due to the fungus *Thielaviopsis ethaceticus*, a wound parasite, often introduced in the ends of plant cuttings. The inside of the stalk becomes red, then black, and gives off a pleasant odour of pine apples. The control of the various diseases is secured by the use of immune varieties, the use of only sound, healthy canes for planting, the isolation of diseased areas and their treatment with lime, the use of fungicide washes, as Bordeaux mixture, and attention to drainage and healthy soil conditions.

**Extraction of the Juice.**—When the cane reaches maturity it loses all its leaves except a wide-spreading bunch at the top, and the stem becomes shiny and of a

lighter colour; the sugar content is then at its maximum. The canes are cut by hand close to the ground and the soft top joints removed with the cluster of leaves, and the stem is cut into pieces about three or four feet long. These are then transported to the factory to be worked up immediately lest deterioration of the juice should occur. In large central factories where canes from outlying plantations are brought, or in cases where a breakdown happens, this cannot always be done, and the canes may remain several days after being cut before they can be crushed. In such case they should be covered with trashed leaves kept moist to prevent desiccation. It has been found that an enzyme present in the tops and capable of diffusing downwards in the stem is liable to cause rapid deterioration of the sugar in the juice, and this supplies a reason for cutting off the tops at once.

**Cane Mills.**—Earlier forms of mills for extracting the juice consisted of two or three rollers of wood, stone, or iron, supported vertically in a strong wooden frame. To one roller a long horizontal lever was fixed, turned by hand or by animal power, and the canes were fed in by hand, the pressure causing the other roller to revolve. In this way only about half of the juice present was extracted. The first step in improvement was to arrange the rollers horizontally, the advantage obtained being that the feed could then be utilized over the whole length of the roller, bevel gearing being used to change the motion. In backward or primitive countries such mills are still in use, driven by wind or water power, but in all more modern factories steam is universally employed as motive power, and several are now using electrically operated mills. The unit mill consists of three rollers placed horizontally one above and two below with a space between, the most effective position being such that the centres of the axles form a triangle with a vertical angle about 80°. The first of the two lower rolls is the feed roll, the other is the megass roll. Between these is placed the dumb turner or megass turner to carry on the crushed cane to the megass roll and prevent

it from becoming buckled in the passage. The adjustment of this turner is a matter of great importance in the efficient working of the mill, ensuring that the crushed cane is passed to the space between the back roll and the upper one in a state that the juice is not reabsorbed and yet without overmuch resistance. The second pair of rolls are placed closer together than the first pair, since the felt of cane passing between them is thinner, owing to the crushing it has undergone in passing through the first pair, and has also lost juice. In a mill of this kind about 60 per cent. of juice is expressed in the double crushing to which the cane has been subjected, or in the most powerful mills 65 per cent. may be expressed. Thus in cane containing 10 per cent. of fibre 25 per cent. of juice still remains in the crushed bagasse. However, as the fibre in its natural undried state contains colloidal water the remaining juice will not have the same density as that already extracted. By passing the bagasse through a second and third mill a further quantity of juice may be extracted, and this is still further increased by the addition of water in restricted amount so as not unnecessarily to dilute the juice.

**Maceration and Imbibition.**—In the addition of water to the bagasse between the first and second mills or between the second and third, it may be done by spraying over the layer of bagasse, when the operation is properly called imbibition, or by passing the bagasse through a bath of water, when it is called maceration. To secure the best results with imbibition the pressure of the mills must be regular and constant, the bagasse well subdivided as it leaves the rolls and fed uniformly; the imbibition must be evenly distributed over the whole surface. After crushing, bagasse absorbs water readily and swells up. As the cell-walls of the juice cells have been broken the water penetrates and dilutes the juice, although the mixing is never complete. It is an advantage to carry out the operation in two stages, the more dilute juice from the third mill being often used instead of water between the first and second mills if the state of the bagasse is such

as to enable it to absorb the juice. If we suppose an extraction of 72 per cent. by the time the bagasse leaves the second mill, it will still contain 18 per cent. of juice, assuming the fibre to be 10 per cent. Now, on the addition of 15 per cent. of water calculated on the original cane, the bagasse will then contain $18+15=33$ per cent. of diluted juice. If this 15 is squeezed out in the third mill there is recovered $15\times\frac{18}{33}=8{\cdot}2$ per cent. of juice. As the mixing is incomplete the actual recovery is less than this. There are limits to the amount of water that may be used for imbibition, since it is necessary to balance the economy of extra sugar gained as juice against the increased cost of fuel required to evaporate the more dilute juice. Further, with repeated imbibition and crushing, impurities are extracted from the fibre, especially where the water added, is rendered alkaline with lime to avoid fermentation. The juice is also more difficult to clarify. The increased yield of juice, moreover, is not proportioned to the water added, and must be kept within limits. The gain, however, is more marked with cane of a higher content of fibre.

In order to render the bagasse more capable of absorbing the water of imbibition and to facilitate crushing, it is now usual to subject the cane to a preliminary cutting or shredding before it is passed through the mill. The cane cutter or shredder is particularly useful when dealing with canes that are bent and do not lie evenly. It consists of a series of slightly curved knives arranged helically on a horizontal axis near the feed-plate of the mill. The knives tear or slit open the canes. The Krajewsky crusher consists of two steel rolls with zigzag corrugations which mesh into each other and break the cane before it is passed through the mill. These arrangements materially increase the amount of cane dealt with and give increased extraction.

**Multiple Crushing.**—Although various forms of mill have at times been tried, the modern form of mill consists of a number of units of the three-roller type preceded or not by a two-roll crusher, thus giving mills with eleven, fourteen, or seventeen rolls. The higher pressing thus

obtained give more of the juice from the bark and nodes of a lower quality than that from the pulp, thus the higher the extraction the lower the quality of the juice. If the purity of the juice is 90·6 with an extraction of 87 per cent., it would be only 89·3 purity when the extraction reached 92 per cent. The sugar obtained from a given juice does not increase proportionally with the extraction; the result

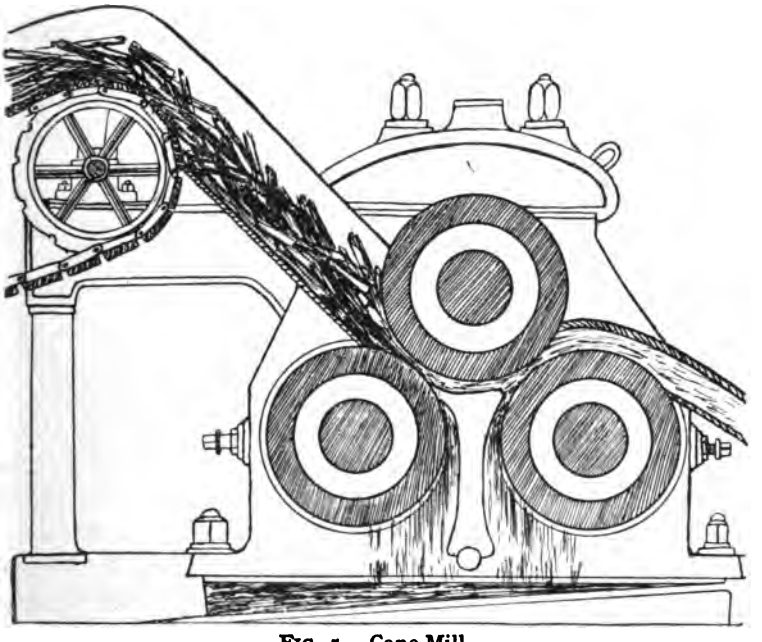

FIG. 5.—Cane Mill.

of the higher extraction, however, is found to be economical, the present mills with multiple units yielding a greater quantity of sugar of slightly lower quality.

**The Diffusion Process.**—Since the cells of plants are able to take up their nutritive materials from outside to inside by purely osmotic processes, it should be possible under suitable conditions to make them yield these up again from inside to outside. Each cell is formed of the cellulose wall, a layer of protoplasm, the cell juice, and the nucleus. The juice cannot leave the living cell because

the protoplasmic envelope is impermeable to the cell juice. It is necessary for the process of extraction of the juice to kill the protoplasmic layer, or at least to modify it by heat, so that it no longer offers an obstacle to the diffusion of the contents, but allows of an osmotic exchange, through the cell-wall, of the dissolved substances—sugar, salts, albumen, colouring matters, etc.—just as through any other membrane. This is the basis of the process of diffusion for extracting the sugar juice from the beetroot, which was successfully applied by Jules Robert at Seelowitz in Moravia in 1864.

At this period double milling of the cane was the rule, and only 83 per cent. of the total sugar present in the cane was extracted, or 86 per cent. when maceration was also used, and attempts were soon made to apply the diffusion method to the cane with the object of increasing the extraction yield. Although the process necessarily entails increased dilution of the juice up to 25 per cent. on the original cane juice and consequent increased expenditure of fuel for evaporation of the extra water, an extraction of 95 to 96 per cent. of the total sugar was obtained. The effect of this increased extraction was to stimulate improved crushing by the use of multiple mills, the adoption of cane breakers or crushers and carefully controlled maceration, whereby the yields by milling were so increased that, all points considered, diffusion no longer offered any advantage. On the other hand, combinations of the two processes have been put forward; cane bagasse, from which part of the juice has been extracted by milling, is put through the diffusion process for the more complete exhaustion of its sugar content. In the diffusion process the cane is cut into thin slices or chips one millimetre or more in thickness, and the chips then filled into a series of 12 or 14 vessels, the diffusers, and treated until sufficiently exhausted. Two of the diffusers are always out of the circuit, one being emptied and the other being filled with fresh chips. Of the remaining twelve, the one that contains the most nearly exhausted chips is supplied with

water to remove the last traces of juice. The dilute juice from this passes through the next, and so on in turn through each cell of the battery, finally passing through the diffuser last filled with fresh chips from which the juice is drawn off for treatment in the factory. The usual course of the circulation in the diffusers is from above downwards, but in the freshly filled diffuser (A, Fig. 6) this direction is reversed, the juice entering from below so as to drive out the air from the chips through the air-cock in the lid cover. As soon as juice issues from this vent, the cock is closed and the direction of flow reversed to the normal and the juice drawn off below. Even by this means it is not

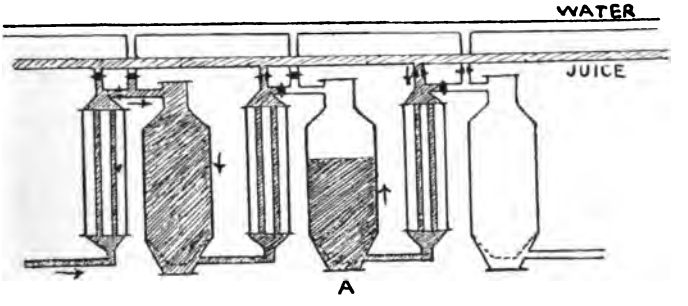


FIG. 6.—Diffusion Battery.

possible to get rid of all the air, and circulation in the battery is thereby considerably impeded. It is also usual to introduce water below in the diffuser containing nearly exhausted chips so as to loosen these and facilitate their removal when the diffuser is opened, because there is a tendency for them to become tightly packed owing to the pressure under which the battery is worked. To each diffuser there is attached a juice heater by which the juice passing from one diffuser to the next is heated during its passage through a series of tubes heated by exhaust steam. By this means the diffusers are maintained at the temperature most favourable for diffusion, usually 85° to 95° C. The diffusers containing the more recently filled chips are quickly raised to 90° C., and this is kept up through the

battery, only falling again at the end containing the nearly exhausted chips. Each diffuser contains a perforated false bottom to prevent the chips being carried through with the juice, and a perforated plate in the upper part serves a similar purpose (Fig. 7). Diffusion juice is purer than mill juice, contains less organic matters in suspension, and is less coloured, colloidal impurities being left behind in the cells; the disadvantages are that it is difficult to cut cane chips, these also tend to pack in the

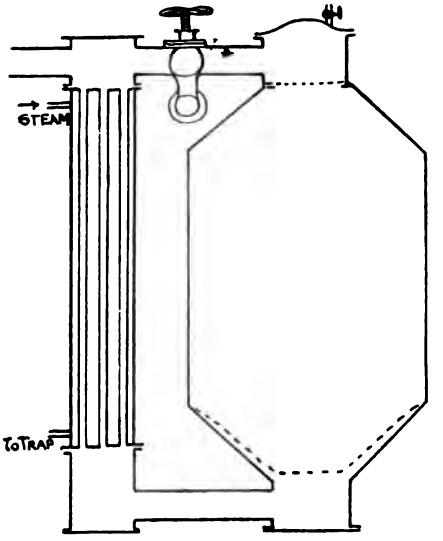


FIG. 7.—Diffuser and Juice Heater.

diffusers and cause bad circulation and consequent fermentation of the juice. The exhausted chips do not form so good a fuel as bagasse, and the juice obtained is more dilute, necessitating an increased consumption of fuel for the extra evaporation. The process also requires skilled supervision, and does not afford so great a margin for a rush of work as mills.

The Naudet process is a combination of milling and diffusion, bagasse from the second mill after dry crushing being taken from which 80 per cent. of the total juice has

been extracted. The bagasse is subjected to diffusion under forced circulation, a pump drawing off the dilute juice from a diffuser and delivering it through the heater to the top of the following diffuser, any particles of loose bagasse or cush-cush are retained by an intervening strainer. The method of mashing or starting a freshly filled diffuser is to pass through the bagasse some hot juice from previous diffusion so as to raise the temperature to 100° C.; mill juice corresponding to the bagasse, limed and sulphited is then pumped through the heater into the diffuser so as to drive out the more dilute juice, and the diffuser is then connected with the battery. By this means the juice drawn off is obtained defecated and filtered.

In the Kesseler process the extraction of sugar from bagasse is continuous, an upright U-shaped pipe being used as the diffuser. The bagasse is forced through the pipe in a compact mass by a screw conveyer, water being forced through at the opposite end of the pipe, and juice leaving where the bagasse enters. After the bagasse has passed through the water it is subjected to great pressure by a screw and discharged from the pipe.

*Megass.*—Megass or bagasse is the refuse of cane after crushing in the mill. The amount of this material to be dealt with is very large. A cane containing 15 per cent. of sugar has a fibre content of 10 or 12 per cent., and the yield of moist or green megass amounts to one-fourth of the weight of the canes worked. The average percentage composition of megass from the mills is, fibre 42, sugar 6 to 7, ash 1·5, water 50, the water content varying according as dry crushing or maceration is used, and being much greater in megass from the diffusion process. By its use as fuel it is generally capable of furnishing the whole of the large quantity of steam necessary for evaporation and power in the factory, but specially constructed furnaces are required owing to the bulky character of this fuel. It is fed automatically from a hopper by a mechanical stoker so arranged as to be under pressure to avoid its being carried away by the draught. Contact of the megass fuel with water-cooled

heating surface is to be avoided, otherwise the reduction of temperature causes loss due to incomplete combustion, distillation products escaping unburnt. Heated air supply ensures better combustion and increased efficiency. Where labour is cheap the megass is partly dried by spreading it in the sun, or in other cases it is passed through a drying chamber, where it is heated by the waste furnace gases during its passage on travelling bands. By this means half of the water may be removed and the water content reduced from 50 to 35 per cent., with a corresponding increase in its value as fuel. If the supply of megass proves insufficient for steam requirements it is supplemented by burning trash, wood, molasses or coal. From 4 to 5 tons of megass are equal in thermal value to a ton of coal. The ash of the megass is used as fertilizer on the cane fields, the large amount of potash and phosphoric acid in it being thus restored to the soil. The soft portion or pith of the megass sifted out and mixed with molasses forms the cattle food molascuit. Owing to its length of fibre megass affords a suitable material for the manufacture of strong wrapping paper in localities where it can be replaced at reasonable cost by coal or other fuel. It has also been used as a filtering medium for cane juice, but there is a tendency for it to render the juice gummy, especially when exposed for long to alkaline (limed) juice at a high temperature.

*Yields.*—The highest yields of sugar per acre are obtained in Java and Hawaii, and the lowest in the primitive parts of India and other tropical countries where the canes are not only poor in sugar, but the methods of manufacture are destructive and wasteful. In Java the sugar cane is an alternative crop with rice and other food crops, and the water of the rivers used for irrigation leaves a fertilizing slime rich in potash and phosphoric acid, giving for the whole island an average yield of 42 tons of canes to the acre. In the Hawaiian Islands a still heavier crop is obtained, as much as 100 tons of canes have been obtained from the rich volcanic loam assisted by irrigation, and with the aid of up-to-date machinery the sugar obtained has reached

5·6 tons to the acre in some of the islands. In Cuba a single factory may crush as much as 600,000 tons of cane in the season, and produce 70,000 tons of sugar. In the West Indies about 20 or 30 tons of canes to the acre is the usual crop.

The results obtained in the Bareilly district of the United Provinces of India given recently by Hulme and Sanghi, show the great need for improvement. Java, using modern methods, obtains 110 maunds (the maund is 82⅔ lbs.) of sugar per acre; Bareilly, with modern methods, 64 maunds; and by native methods only 7½ maunds per acre. From these figures it is not surprising that, in spite of a 10 per cent. import duty and the payment of freight, railway, handling, warehousing, and other charges, Java can sell sugar in the interior of India against that produced locally.

*Character of the Juice.*—Cane juice direct from the mill is an opaque liquid, frothy from entangled air, and in colour from grey to yellowish or dark green, according to the cane from which it is derived. It contains in solution all the soluble constituents of the cane; sucrose, reducing sugars, pectin, gums, salts of potash, soda, lime and iron as sulphates, chlorides, phosphates, silicates and salts of organic acids; and in suspension, sand and earth from the soil adhering to the cane, fragments of fibre, wax, and colouring matters. The fresh juice has a faintly acid reaction to litmus, more pronounced when burnt or damaged canes are worked, and is viscous from the presence of albuminoids, pectin, and gums so that it cannot easily be filtered cold.

The colouring matters of the cane are chlorophyll, saccharetin, and, in dark-coloured canes, anthocyan. Neither chlorophyll nor saccharetin dissolves in the juice, but enters it with the cush-cush, as the finely divided megass fragments are called, and is thus present only as suspended particles; but by the action of lime or alkalies the saccharetin turns yellow and passes into solution. The colour of Demerara crystals as originally made was due to this substance. Anthocyan, the pigment of dark-coloured canes, is freely soluble and adds its quota to the colour of the juice. As

Zerban has shown, the darkening of expressed juice, which is colourless while in the cane, is due to the action of oxidase and peroxidase ferments on the polyphenols or tannins, and a green colour is caused by a trace of iron dissolved from the mill rolls by the organic acids present in the juice. A closer study of these reactions will doubtless lead to methods for the removal of the colour or the avoidance of its formation.

*Clarification.*—The turbid juice as it flows from the mill passes through a coarse strainer of wire gauze to remove cush-cush before it is pumped to the measuring tanks. It is then defecated by treatment with milk of lime, usually at 20 degrees Baumé, and passed through a juice heater to enter the defecator or clarifier where the separation of a scum and a sediment takes place, leaving a more or less clarified juice ready to be sent to the evaporators. This tempering with lime is an operation requiring considerable experience; the amount added must be sufficient to neutralize the free natural acidity of the juice and such as is found by trial to give a ready separation of the sediment. By the action of the lime and heat albumen is coagulated, much of the pectin also becomes insoluble, and calcium phosphate forms as a flocculent precipitate. If too little lime is added the precipitate does not settle readily; and if too much, the clarified juice is left alkaline, and on being heated, dark-coloured compounds are formed from the reducing sugars present. Since each separate lot of canes differs in the amount of lime needed, constant adjustments are necessary to keep pace with the varying composition of the juice. Some juices will not clarify without an excess of lime, and the alkaline juice in this case must be neutralised by passing through the sulphuring box, in which it is subjected to a stream of sulphur dioxide, or by the addition of phosphoric acid when a slight excess only of lime has been used. As it is impossible to filter the juice the clarified portion of juice between the scum and the sediment is drawn off as soon as the separation is sufficiently complete, the operation being thus merely a decantation.

*Defecation Agents.*—Besides lime and sulphurous and

phosphoric acids many other agents, either alone or in combination, have been proposed and used for clarification, the object in view being to cause a separation of mechanical and dissolved impurities by the formation of a more or less bulky precipitate, and at the same time to effect a decoloration of the juice. Among these may be mentioned salts of aluminium, zinc, tin, and even lead. Barium hydroxide and salts of barium, tannic acid, ozone, and other oxidising agents and oxalic acid. Many of these are effective enough but from the expense involved, or from their poisonous nature, are either not economical or obviously inadvisable, and in practice only the three agents first mentioned are in general use. Manoury used 0·75 grm. of BaO and 1 grm. of magnesium sulphate per litre of juice after defecation with lime. The filtrate from this treatment was free from the defects of decanted juice. Processes which do not permit of filtration of the turbid juice lead to ready fouling of the evaporators and boiling apparatus, causing loss from the frequent cleanings necessary. The transmission of heat is also retarded by the deposits formed. A dilute solution of formaldehyde was used by Simpson at the Cavadonga central factory in 1906–1907. At a concentration of 1 in 20,000 formaldehyde was found to retard the decomposition of cane juice for several hours. It hardens the tissues and cells of the fragments of cane in suspension in the juice and precipitates them with the scums so that they are eliminated from the juice. It coagulates the albumen which is taken up by the lime and removed, and also precipitates pectic matters and ferments, and thus lessens the tendency to acidification, and it does not combine with sucrose or alter it. The vapours pass off on evaporation and are harmless to the workmen. An increase of purity of the juice thus treated was found of 1·0 to 1·7.

Hydrosulphites are used to bleach the juice, particularly when the object is to manufacture sugars for direct consumption. The sodium salt is known under the name of Blankit and the calcium salt as Redos. These salts act as powerful reducing agents on the colouring matters of the

juice, much more so than sulphurous acid, but as with the use of sulphurous acid and sulphites the flavour of the products is adversely affected, notwithstanding the small proportion required. The reaction proceeds according to the equation: $Na_2S_2O_4+O+H_2O=2NaHSO_4$. The dark products formed by overheating the juice are far less readily bleached.

**Defecators.**—Instead of completing the operation of defecation in one vessel where the juice is limed and heated by steam, the scums removed, the sediment allowed to subside, and the clarified juice run off for concentration, it is now the almost universal practice to heat the juice in a juice-heater to remove air-bubbles and then transfer it to special vessels, the defecators or clarifiers, where lime is added and, after settling, the clear juice is decanted. In Mauritius a form of continuous settling tank called the *Bac Portal* consists of a shallow tank about 15 feet square, divided by parallel partitions into a series of troughs a foot wide. A portion of the partition is removed at alternate ends of the troughs so that the juice entering at one corner of the tank overflows from one trough to another, and takes a zigzag course of about 200 feet, depositing in its passage the bulk of the suspended impurities. In Hatton's continuous defecator the cold limed juice enters in a regular and steady stream at the bottom, and as the level rises overflows into the smaller suspended vessel from which the clear juice flows off as the vessel fills. The juice is heated by steam admitted to the double bottom, to a temperature not exceeding 100° C., the temperature being maintained constant by an automatic regulating device. The scums rise and form a layer on the surface several inches thick. These are removed in a fairly dry state, and need no further

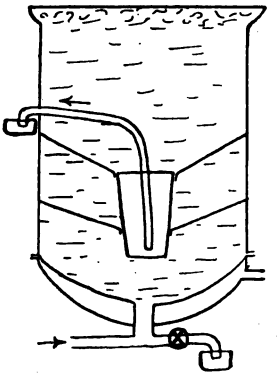

FIG. 8.—Hatton's Continuous Defecator.

treatment as their sugar content is very small. Any sediment which may fall and adhere to the bottom, thus lessening the transmission of heat, is removed by means of a scraper which is gently rotated to loosen the incrustation, and then suddenly opening the bottom discharge cock for a moment, which allows the rush of juice to carry out the deposit. In the Deming system of superheat clarification the impurities are precipitated and removed automatically and continuously. The limed juice is forced by a pump first through the absorber, and then through the superheater, where it is heated to 110°–120° C., and, finally, through the absorber, where it is cooled down to 94° C., by giving up its heat to the incoming cold juice. The quantity of juice required to fill this heating arrangement is small, and during the operation it is under heat but 45 seconds, being discharged at a temperature below that possible in the open clarifier system. It then enters the continuous settling tank from which the juice is constantly decanted to the evaporators. There are no surface scums to require skimming or brushing, everything being precipitated with the mud to the bottom of the tank, where they are drawn off. This system uses but one-half to two-thirds of the amount of lime required by the open process, because lime more readily combines with cold juice, and the instantaneous application of high heat increases the action of the lime during the process of clarification. The muddy juice drawn off below is sent to filter presses for filtration, the plant required being only one-tenth of that which would be required to filter the whole.

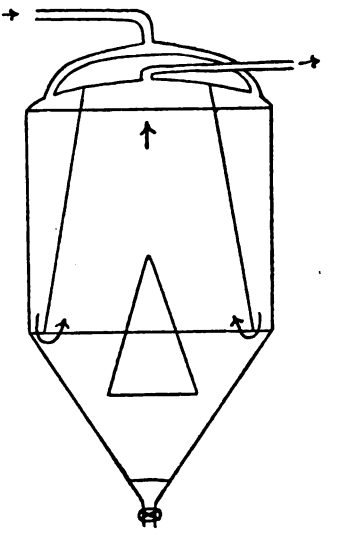

FIG. 9.—Continuous Settling Tank of Deming's Clarification System.

**Sulphuring.**—In the ordinary defecation process only enough lime is used to neutralize the natural acidity of the juice, but with a greater tempering there will be a more energetic action of the lime on the impurities in the juice. The latter would in this case be left alkaline, and to prevent the alkalinity from affecting the juice adversely, the lime is removed with sulphur dioxide. To each 1000 litres of juice about 10 litres of milk of lime at 15° Baumé are added, and the limed juice is treated with sulphur dioxide either by pumping the gas through a perforated pipe lying in the tank or, more usually, by means of a sulphur box. In this the juice enters at the top and falls in a shower on a series of trays, while the sulphurous acid gas, generated in a sulphur oven, is injected below by a jet of steam, the supply of gas being so regulated that the issuing juice is neutral to phenolphthalein. The turbid juice is then passed through a heater so as to raise its temperature to 90° C., and then into a defecation tank where it is brought to the boiling-point—102°–104° C., and the precipitated calcium sulphite and other impurities allowed to settle, and the clear juice drawn off to be concentrated. In the West Indies the juice is "sulphured" first while hot until the colour becomes lighter, due to acidification rather than to bleaching, and a decided separation of flocculent matter takes place, and then milk of lime is added until the juice is neutral. The slightly opalescent subsided juice is then sent to the eliminators. The action of sulphurous acid prevents fermentation, decolorizes and effects a coagulation of those albuminous matters which are not affected by heat alone.

**Eliminators.**—In well-appointed sugar factories the clarified juice is subjected to a further purification before being admitted to the multiple effect for evaporation. The tank used for this purpose is called an eliminator, and is provided with heating coils or pipes, and a gutter into which the frothy scum overflows. The contents of the tank are brought to violent ebullition, phosphoric acid added, and the boiling continued for some minutes; meanwhile the impurities are brushed away with the scum or overflow by

the circulation of the juice until the contents are left clean.

**Carbonatation.**—For the manufacture of a superior quality of refining crystals, or of plantation white sugar, the process of carbonatation is employed in Java and in Queensland. By this means all the beneficial effects of heavy liming are secured in removing gums and other organic impurities without the injurious effects on the reducing sugars which otherwise ensues from the application of high temperature in presence of strong alkalinity, and the whole of the juice is filtered, thus yielding a perfectly clear juice. From 1 to 1·5 per cent. of lime is used as milk of lime, the temperature while alkaline being maintained at not over 50° C. At this temperature the effect of the lime on the "glucose" is to produce lactic acid, the salts of which are colourless; at higher temperatures other compounds of a dark colour would be produced. Carbonic acid is then pumped through the juice until the alkalinity is reduced to the desired point, and the whole is then filtered through filter presses. During the saturation with carbonic acid the temperature is raised to 90° C. The saturation may be completed in one operation called single saturation, when the gas is passed in until neutrality is reached, or in two stages, double saturation, in the first of which the alkalinity is reduced until equal to 0·04 per cent. of CaO, when the juice is filtered and then again saturated until neutrality is reached. Although in double saturation the juice remains much longer in an alkaline condition than with single saturation, and suffers somewhat in consequence, the reason for its adoption is that it is less liable to result in the impurities being redissolved, since these are only insoluble while the juice remains alkaline. The lime added to cane juice in the first place combines with and neutralizes the free organic acids and forms corresponding calcium salts. Next, any alkali salts of acids which are capable of forming insoluble compounds with the lime will be decomposed and the alkali will be set free as hydroxide. Part of the alkalinity, therefore, produced on adding lime is due to potassium or sodium

hydroxide. Further, the lime combines with the albuminoids, gums, and pectins, as well as with the colouring matters, and forms a precipitate with these various bodies which remains insoluble so long as the liquid remains alkaline. Moreover, lime also combines with sucrose to form soluble sucrate of lime so that the reaction which takes place during neutralization is not nearly so simple as that which occurs on passing carbon dioxide into lime water or milk of lime, the simple precipitation of calcium carbonate followed by the formation of some soluble calcium bicarbonate when excess of the gas is used. The saturation gas from the kiln, cooled with the water used for washing it, and containing about 30 per cent. of carbon dioxide, when pumped into the limed juice is rapidly absorbed at the commencement, the escaping gas containing only about 3 or 4 per cent. of $CO_2$. As soon, however, as a certain stage of carbonatation is reached the liquid is observed to become thick and viscous, the rate of absorption of the gas falls off, and the whole contents begin to froth up. This is due to the formation of calcium hydrosucrocarbonate, $C_{12}H_{22}O_{11}2Ca(OH)_2.3CaCO_3$. The supply of gas is now decreased by connecting up to the next saturation tank and the froth beaten down by a jet of steam, and as the liquid becomes heated the insoluble hydrosucrocarbonate gradually decomposes into $CaCO_3$, and sugar and the carbonic acid is again more readily absorbed and the operation is continued until the desired alkalinity or neutrality is reached. In order to avoid the formation of soluble calcium bicarbonate the saturation is stopped when the reaction to phenolphthalein paper is only a faint pink. At this point all the lime has been precipitated as carbonate ; the faint alkalinity is due to potassium (and sodium) carbonate.

**Treatment of Scums.**—The skimmings and muddy residual juice from the various defecation operations are usually run into a tank, mixed with lime, heated and allowed to settle. The clear liquor is decanted and mixed with the bulk of the clarified juice, while the residue is passed through Taylor filters (bag filters) or put through filter presses.

These turbid juices should be worked up as quickly and as hot as possible, owing to their increased liability to fermentation. In filtering, the pressure should be maintained steady. When great difficulty is found in getting a firm cake, as occurs when unripe canes are worked, the remedy is to add a solution of 1 per cent. oxalic acid, and then lime, until distinctly alkaline before filtering; or kieselguhr may be used. The defecation juice gives, as a rule, 90 per cent. of clear liquid and 10 per cent. of muddy liquid from deposit and scums. As an alternative to treatment the scums may also be used to feed cattle, and in those countries where a distillery is run in connection with the factory they are fermented to make rum.

**Filtering Media.**—Filter cloths soon become coated with a thin, slimy film of the suspended matters, which completely stops filtration, so that various substances have been tried with the object of obtaining a dry, firm cake in the filter press. Bagasse has been used, but has a tendency to give the juice a gummy consistency. Peat, lignite, kaolin, or China clay and diatomite, have all found more or less application for this purpose. Sand filters of various forms are also used. In Hawaii wood fibre called "Excelsior" has proved satisfactory.

**Evaporation.**—The clear yellowish juice after defecation is usually about 10° Baumé, requires now to be concentrated to a liquor of 27° to 30° Baumé, suitable for boiling to grain in the crystallizing process.

**Earlier Methods.**—In the tropics where the grower crushes his own canes, the vessel in which the juice is heated serves both as defecating tank and evaporator. It is heated over the fire and the concentration is carried through to form a syrup which when cooled forms a mass of syrup and crystals of sugar. Working on a larger scale the single pot was replaced by several cauldrons of copper built on brickwork and set so that the fire under the first copper supplied heat for the following vessels:—

**Copper Wall or Battery.**—Thus we reach the stage which is still in use in some of the West India Islands. The juice

as it becomes concentrated by boiling is ladled from one copper to the next until the required density is reached, according as it is to be boiled to a high-class sugar in a vacuum pan, when it is withdrawn at about 20° Bé, or to a dense syrup when muscovado sugar is to be made. The high temperature to which the juice is exposed and contact with the air results in a considerable loss of sugar. Various modifications have been made in the arrangement with a view to avoiding the losses. Instead of placing the first or large copper directly over the fire, the last, containing the most concentrated juice, is given this position. The vessels have also been arranged in cascade form to obviate the necessity of ladling the contents which in this case are run from one vessel by a cock to the one next below.

**Panela Train and Fryer's Concretor.**—In order to increase the surface of contact the pans are replaced in the Panela concretor by a long shallow tank divided by numerous transverse partitions, with openings at alternate opposite sides so that the juice traverses in a tortuous course the whole length of the tank in passing towards the end where the fire is placed, and from which the syrup is drawn off in a state sufficiently concentrated to fill into moulds to set. In 1865 Alfred Fryer, a Manchester sugar refiner, introduced his concretor by which he sought to thicken the juice as quickly as possible and at minimum cost to a solid condition—concrete—suitable for transport. The clarified juice runs in a thin layer over a long sloping platform formed of iron trays placed over a flue, and the juice is caused to pass from side to side in its descent by partitions so as to travel a long distance, and the bulk of the evaporation is thus effected. The evaporation is completed in a rotating hollow cylindrical vessel provided with scroll-shaped plates which lift the partially concentrated juice from the lower part in which a supply is maintained, and expose it to a current of heated air passed through the cylinder by a fan. After a short time the syrup is drawn off, cooled, and then forms a solid mass ready to be shipped for refining.

The next improvement was the use of steam as heating

agent, thus avoiding excessive overheating. This was applied to the various forms of evaporators already enumerated.

Another method of applying the advantages of film evaporation is exhibited in Wetzel's evaporator, one form of which was a horizontal helical coil of copper steam piping, revolving so that the lower part dipped into the juice contained in a trough, and in its revolution carried up a thin layer which rapidly evaporated.

Bour's pan consisted of a series of hollow lenticular drums to the interior of which steam was admitted. The edges of these likewise dipped into a trough of juice, and on revolving evaporated the adherent film of juice. Aspinall's pan is a cylindrical vessel in which is a calandria or drum provided with a number of brass tubes through which the juice in the pan can circulate. High-pressure steam is admitted to the drum around the tubes and the juice is thus concentrated. All these forms of evaporator expose the juice to the atmosphere and to high temperature with the danger of injuring the juice.

**The Vacuum Pan.**—The introduction of the vacuum pan in which the boiling temperature is reduced and where the juice is concentrated out of contact with the air, was the first advance towards remedying the disadvantages of open evaporation. The early form of vacuum pan invented by Howard in 1830 was a globular vessel furnished with a coil of piping for heating the contents of the pan by steam. The vapour from the syrup passed by a wide neck at the top of the pan to the "save-all," where any spray carried over strikes against a pipe and trickles down to be returned to the pan, while the vapour passes over and enters the pipe to meet a jet of water whereby it is condensed. This pipe is connected to the air-pump which creates the vacuum by pumping away air and any incondensible vapours. Owing to the costly operation of the vacuum pan as regards fuel consumption the bulk of the evaporation is carried out by the copper wall or other evaporators and the vacuum pan used to complete the boiling of the thickened juice to sugar.

**Triple Effect.**—The preliminary evaporation, however,

is now done in all modern factories by multiple vacuum evaporators usually three in number, although quadruple effects are common, and even quintuple effects are in use. The first vessel is heated by steam, and the vapour from the boiling juice in this heats the second, and so on. The principle of multiple evaporation with a vacuum was first applied by Norbert Rillieux, a French engineer in Louisiana, in 1834. The triple effect consists of three cylindrical vessels with dome-shaped heads with a wide neck which carries the vapour from the juice from one vessel to heat the next.

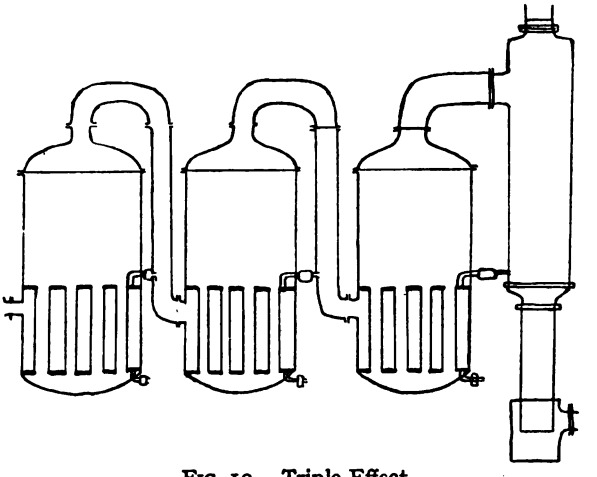

FIG. 10.—Triple Effect.

In the lower part of each vessel is the calandria or steam drum formed by two plates fitted with vertical copper tubes, through which the juice can circulate from the lower part of the vessel to rise just above the top of the calandria, and as it boils and thickens the vapour passes into the calandria of the next vessel. The juice in this makes it act as a surface condenser and assist the boiling in the first vessel, while the vapour of the second vessel enters the calandria of the third vessel, which is connected to the vacuum pump. The steam supplied to the first vessel thus brings about an evaporation nearly three times what it would do in a single vessel, and secures the economy in fuel which has made the

use of the triple effect practicable. There is a pipe to lead incondensible gases from the calandria to the vapour pipe so as to prevent the steam space becoming air locked, and another pipe below to lead away condensed steam or vapour to steam traps. There are also (not shown in the Fig.) pipes leading the juice from one effect to the next. By means of multiple effects one pound of steam could evaporate an unlimited quantity of juice, but for practical reasons the limit is admittedly generally reached with quadruple effects. The evaporation is due partly to the direct steam used and partly to the heat from the juice itself which enters hot and leaves the last vessel at a lower temperature. At the same time the latent heat of vapour is higher as the temperature of vaporization is lowered and the losses from radiation have to be allowed for.

It is generally stated that one pound of steam evaporates 1·8 lbs. of water in a double effect, 2·7 lbs. in a triple effect, and 3·5 lbs. in a quadruple effect, but these figures will necessarily vary with the conditions of the evaporation, besides which the steam used in working the vacuum and other pumps must be taken into account.

**Lillie and Kestner Evaporators.**—There are various forms of evaporator with horizontal heating tubes in some of which the steam is inside, in others outside, the tubes; one arrangement of this kind which differs materially in mechanical detail is the Lillie evaporator. The characteristic feature of this system is that it is a film evaporator. The tubes are expanded into the tube plate at one end where the steam enters, the other end being free and closed except for a small orifice to allow of the escape of air which finds its way into the vacuum space. The juice is circulated by a rotatory pump and overflows from a series of distributing troughs in a shower over the tubes which are staggered in vertical rows to ensure a perfect film over all. This apparatus offers special facilities for cleaning both the inside and outside of the tubes, and there is no thick layer of juice through which the vapours have to force their way so that evaporation is greatly facilitated. A portion of the

circulating juice is withdrawn continuously while fresh juice is supplied to make up for what passes off as vapour and in the discharge.

The Kestner evaporator is another essentially different form of apparatus by which a more uniform film of juice over the entire tube surface is obtained than in the preceding horizontal effect. In this apparatus the calandria is placed in a vertical position, and steam is admitted to the outside of the tubes. Juice from the supply tank (Fig. 11) enters the tubes through the valve V, while steam is admitted at A; B is connected to the vacuum pump, and the concentrated juice is drawn off by a pump at L.

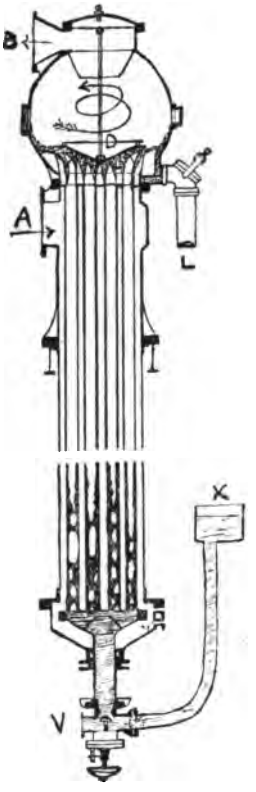


FIG. 11.—Kestner Evaporator.

A climbing action ensues as soon as the liquid begins to boil, the vapour generated filling the centre of the tube and carrying up with it a film of the liquor which climbs up the whole length of the tube. At D is a centrifugal separator to prevent the concentrated juice being carried away by entrainment. The juice is a minimum of time under heat, and the coefficient of transmission of heat is high. Both the Lillie and Kestner evaporators are usually worked as multiple effects.

**Boiling.**—The juice from the triple effect is usually a syrup of 30° Bé, and contains about 54 per cent. of solids in solution, but has become turbid owing to some of the lime salts and other impurities having become insoluble in the more concentrated solution, and before being passed to the vacuum pan to be boiled to grain is frequently further treated either by simply allowing the syrup to stand while the suspended matter subsides, or it is sulphured or phosphoric acid added,

and then mechanically filtered. In the vacuum pan the charge of syrup is boiled until the solution of sugar becomes saturated at the temperature prevailing in the pan, and then the temperature is caused to fall either by shutting off the supply of steam or by increasing the flow of water to the condenser and thus increasing the vacuum.

**Crystallization.**—The sugar then separates in minute, scarcely visible crystals. Instead of graining the pan, as this is called, the addition of small grained sugar is sometimes made as seed to the saturated syrup in the pan. The next step is to grow the crystals by drawing in a further supply of syrup and continuing the boiling. The points to be attended to are that the production of a second crop of crystals, called false grain, is to be avoided so that there must be no sudden lowering of temperature allowed, but the fresh charges of syrup are to be drawn in from time to time as the crystals grow. Should false grain appear, the small crystals are washed out, that is, dissolved, by drawing in a larger charge of syrup or by lowering the vacuum and thus raising the temperature. When the pan is sufficiently full the charge is closed or brought up to strike by gradually raising the temperature until the magma of syrup and crystals, called the massecuite, is ready to be dropped. When larger crystals are required it is sometimes done by cutting the pan, as it is called—that is, by dropping half the charge and then continuing the boiling until the pan is full again.

**Muscovado Tanks.**—The concentrated syrup from the last pan of the copper wall or other open evaporator, owing to the high temperature, does not contain crystals. In order to start crystallization it is drawn off into crystallizing tanks where, on cooling, a crust of crystalline sugar forms. This is broken from time to time to help the cooling until, finally, a magma of fine crystals and molasses is obtained. Better crystals are obtained by the use of muscovado tanks. These are hemispherical troughs over which is an axis bearing several wooden arms which are oscillated in the mass during the cooling of the syrup assisted by the stirring, so that the sugar, instead of forming a larger number of small

grains, gradually deposits on the crystals already formed. This quality of sugar, although of low grade, has a rich flavour for which it is esteemed.

**Vacuum Pan.**—Instead of the small and more or less globular vessel formerly used, vacuum pans are now made of enormous size to cope with the large amounts of juice dealt with in modern factories. The form adopted as most suitable is a cylinder, the lower end is conical and fitted with an outlet valve of large aperture. There are many coils so arranged that steam may be admitted to the lowest one first, and to the others as they become covered with syrup. This arrangement is to prevent splashes of syrup being caramelized, as they would be if projected on to a hot, dry coil. The dome of the pan is provided with baffle plates to prevent spray from being carried over with the vapour and consequent loss of sugar by entrainment. There are also light and sight glasses to enable the panman to observe the boiling mass, and a proof-stick to enable him to withdraw samples without breaking the vacuum. In these samples, which he spreads on a glass plate, he can watch the progress of the crystallization and note whether false grain is beginning to form and if the crystals are otherwise satisfactory. His aim is to obtain crystals all of uniform size, since these part more readily with the mother liquor when spun in the centrifugal, and any small grain present may be dissolved and pass into the syrup and thus be lost to the first product.

**Curing.**—The earlier method of treating the mass of sugar and syrup obtained by boiling in open pans, so as to produce a sugar more or less free from adhering molasses, was by the process of claying. The hot mass was poured into conical earthenware moulds, the opening at the tip being closed with a wooden plug. The moulds were arranged point downwards until the mass set; at the end of 18 to 20 hours the plug was removed and the mould placed over a pot to drain. The pots were replaced after another 20 hours by fresh empty pots, and a puddle of wet clay was placed on the sugar at the base of each mould. The water in the clay slowly filtered out, and spreading in the mass of sugar

carried with it the viscous layer of molasses adhering to the crystals, for the syrup is more readily soluble than the sugar crystals. When the first portion of clay was dry it was replaced by another, and often by a third, until the sugar was sufficiently white and purified. It was then dried in a stove, broken into coarse powder, and packed for shipment abroad. The whole operation took some weeks and required very considerable space, and the method is now almost completely replaced by the process of treatment in centrifugal machines which, moreover, has rendered possible the boiling of sugar in the vacuum pan. Massecuite from the pan is dropped into a receiver called a mixer, where it is stirred to keep the magma uniform, and portions drawn off while still hot into centrifugals. There the molasses is spun off, then water, steam, or purer syrup, called clairce, sprayed on the surface until the sugar is purged sufficiently. The molasses and washed-off syrup are kept separate, since the latter is of richer quality, and these are submitted to further treatment.

**Crystallizers.**—Instead of drying the massecuite hot as described above it is, especially if of a low-quality product, run into a crystallizer, where it is kept stirred, the temperature meanwhile being allowed to fall gradually under control by the circulation of water or steam in the jacket. This is continued until the mass has cooled sufficiently for the crystals to take up all the sugar possible and the mother syrup is exhausted, but the mass is still warm enough to be treated in the centrifugal machine. During this cooling, as crystallization proceeds, and the mass becomes colder, it is necessary from time to time to add a portion of exhausted molasses from a previous operation to keep the mass sufficiently fluid, otherwise it would become too thick to work in the centrifugal. The crystallizers are usually large vessels, one or two of which are capable of holding the whole contents of a pan. They are either semi-cylindrical troughs or cylinders provided with powerful stirring arms which make one revolution in about two minutes. After this crystallization in motion the molasses is obtained in a more completely exhausted condition, owing to the sugar having

crystallized in a separable form by deposition on the crystals already present during the fall in temperature.

**Centrifugals.**—The centrifugal machine consists essentially of a drum called the basket with perforated walls, which is rotated at a great speed. Inside the drum is placed the lining or cloth, a sheet of perforated metal which fits against the inner surface of the wall. When the massecuite is allowed to flow into the drum, while the latter is rotating slowly, getting up speed, it is thrown against the side until a sufficient charge is in, and the speed having now increased the syrup is thrown off and passes through the perforations against the wall of the casing surrounding the drum, from which it drains down into a gutter around the inside of the base of the casing and flows away. The syrup is boiled again for a second quality product, while the richer syrup produced by washing the charge in the machine is usually returned to be boiled in the next batch of first product. When the machine is stopped the sugar is removed through an opening in the bottom of the drum.

**Molasses.**—The syrups obtained from a massecuite in the centrifugal are spoken of as first or second molasses, and so on, according as they are derived from first or second product, but when the term "molasses" is used without qualification it is exhausted molasses that is meant—that is, a syrup from which no more sugar can be obtained by boiling and otherwise dealing with it in the usual way. Cane molasses, however, still contains from 25 to 40 per cent. of sucrose, and from 5 to 30 per cent. of glucose or reducing sugars. The reason why all this sugar remains immobilized in this exhausted syrup is to be traced to the combination formed by the sugars with the various salts present in it into an uncrystallizable compound. After molasses has been boiled as thick as possible it cools to a blank, vitreous mass. Further details on molasses will be found in the section on beet molasses.

**Composition.**—Exhausted molasses considered as the mother syrup from a final massecuite varies greatly in composition dependent on the methods of treatment during

manufacture and on the purity of the original juice. The amount of sucrose held in solution in the water present is less than that in a saturated solution of pure sucrose at the same temperature, but the sum of sucrose and reducing sugars is greater than that of pure sugar. It is not practicable to boil cane syrups as close as those from beetroot, owing to their greater viscosity, for otherwise they would not work in the centrifugals. The moisture of the undiluted molasses is usually about 15 per cent., but this is raised to 20 per cent. due to the water used in purging the sugar. The sucrose may be from 30 to 40 per cent., glucose 15 to 25, ash 7 to 10, water 16 to 20, organic matter 12 to 15 per cent.

**Utilization.**—The economical disposal of molasses offers an important problem for solution. The immense output of the product represents a loss of 8 per cent. of the sugar in the cane, yet the low price it commands and the cost of carriage, notwithstanding its large content of sugars, do not secure a sufficient outlet for its complete utilization, so that in some cases it is run into the nearest stream to get rid of it. The small quantity used directly as food is insignificant, but cattle consume it readily owing to its attractive flavour, and from its richness in nutritive material they fatten upon it and improve in condition. Carbohydrates favour muscular work, but the poverty of molasses in nitrogenous foodstuff makes it necessary to use it mixed with oil cake. Its fluid condition, however, presents difficulties in transport and in doling out the quantities as required. This objection is removed by the use of absorbent material to soak it up in the same way that peat is used with beetroot molasses. At the cane factory a suitable material is found in the softer part of the bagasse—the pith—the cellular tissue of which originally held seven or eight times its weight of juice, and will absorb four times its weight of molasses and give a material which does not stain the bags in which it is packed, and the cellulose is also in great part digestible. In some cases enough of the pith may be collected below the bagasse carriers, where it has worked through the slats, or the bagasse is ground and sifted. It is then dried and mixed

with the hot molasses to form the cattle food called Molascuit. Another food is made with the millings of rice (paddy), but this material is not so good an absorbent as the pith. Molasses is also used as fuel. When sprinkled on bagasse and burnt it tends to form a compact coke and choke the fire, and at a higher temperature the alkali present combines with the silica of the cane to form a glassy slag difficult to remove from the fire bars. It may also be burnt by spraying in the same way as oil, but this requires a special form of furnace. The ash contains a large proportion of potash, and is, therefore, useful as a fertilizer, indeed, diluted molasses may be spread on the fields as a manure when the acidity it produces is beneficial in destroying the nematodes which injure the canes.

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## Section II.—BEET SUGAR

**The Beetroot.**—The raw material for the manufacture of sugar in Europe and other lands with temperate climates is furnished by the white sugar beet derived from the wild *Beta vulgaris* or *B. maritima* found growing in salt marshes and belonging to the order Chenopodiaceæ which includes the goose-foot, spinach, and saltwort. Various sports when cultivated become fixed in character and differ in size, form, colour, foliage, and richness in sugar, as the Vilmorin, Quedlinbourg, Wanzlebener, Imperial, etc. Originally an annual, it has by cultivation become a biennial; grown in the first year from seed it forms only root and leaves, and, on replanting, or after remaining in the ground throughout the winter, in the next season it bears a stalk with flowers and seed. Occasionally, however, beets run to seed during the first year, but the roots are then woody and small, and although still rich in sugar, are difficult to cut for use. The characters of a good sugar beet are a conical, somewhat pear-shaped, tap-root, almost completely buried in the soil, the more so the richer it is in sugar, for the green portion or neck which rests above the ground contains a lower percentage of sugar. The skin is rough to the touch and has a longitudinal groove on each side, with well-developed hairy rootlets which penetrate deeply into the soil. When the soil is stony or too compact and the rootlets cannot thus penetrate, the root becomes forked, and there is loss in harvesting and the beets are smaller and less rich. Experimental trials have proved that beets rich in sugar can be grown in this country equal to those on the continent, but for various reasons, mainly due to political obsessions, the beet sugar industry has not been established here. Large sums are required to equip a factory for the manufacture of

sugar, and guarantees are necessary to ensure delivery of a sufficient supply of beets to keep a factory working economically, since this can only be done when the work is on a fairly large scale; on the other hand, the deep ploughing and thorough weeding needed for growing sugar beets successfully leaves the soil in a condition that is highly beneficial for other crops to follow.

**Cultivation.**—By proper treatment and manuring any soil may be made fit for growing beetroot, but the best results are obtained with calcareous loams or siliceous clays with calcareous subsoil. A proportion of humus is needed to regulate humidity, as it only gives up moisture slowly in dry weather, but an excessive amount renders the soil too acid. The land should be ploughed in autumn to a depth of ten inches or more, and farmyard manure used at that time will result in a good yield and quality of beet while its use in spring is injurious to the crop. As soon as the soil is dry in spring it is ploughed, harrowed, rolled, and spread with nitrogenous and phosphatic fertilizers. The seed is sown by drills in rows about 16 inches apart on the level, and not over half an inch deep; about 25 to 35 lbs. per acre is required. The most favourable time for sowing is usually about the middle of April. The plants will be well above ground about a fortnight after, when the ground is hoed by hand and the hoeing is repeated several times until the leaves cover the ground. The rows are then cut through, leaving a space of 8 to 12 inches between the clusters left standing. When these plants have roots as thick as a straw they are thinned by hand, leaving only the strongest plant in each standing. As soon as the leaves quite cover the ground, about the end of July, no further work is done on the field until the harvest. During the months of August to October the sugar is formed and accumulates in the root. The beet is richer in sugar in proportion as the autumn is drier and sunnier. From the middle of August onwards tests are made to determine the sugar content of the roots, and to fix the time for taking them up. This should not be later than the end of October in order to avoid frost. After the roots are taken up and well

cleaned from adhering soil, the leaves and top of each beet are cut off to prevent the stored beets from forcing new shoots, as these would use up the sugar. The harvested beets are either sent direct to the factory or thrown in heaps on the field. If these are to remain some time before being removed they are covered with the leaves to protect them against drying and the night frosts. The roots not immediately used at the factory are stored in silos. The attacks of nematodes, caterpillars, and other injurious pests are kept in check by not planting the same field too frequently with beetroot, a rotation of crops being usual, the beet following every fourth year after wheat or barley. The yield of good sugar beets, freed from soil and trimmed, varies from 10 to 20 tons per acre, and the sugar content from 14 to 16 per cent. Forage beets and those grown for producing alcohol give a greater weight of roots per acre, but the total sugar is no greater and the impurities (non-sugars) present in the juice while they interfere with the extraction of sugar, are no detriment in the production of spirit, and are of alimentary value, the choice of the kind of beet grown being dependent on whether the tax is levied on the weight of roots or not.

**Improvement by Selection.**—Perhaps no other agricultural crop has shown so markedly the importance of seed selection as the sugar beet. By the careful choice of those varieties which seemed most favourable to the production of sugar and the selection of roots for the production of seed during the succeeding year, and by judicious and scientific fertilizing for the purpose of increasing the sugar content without unduly increasing its size, the beet has been placed at the head of the sugar-producing plants of the world. The beets to be used for producing seed (mother beets) should be of regular form, high content of sugar, and purity of juice with other characters. Under similar conditions a beet rich in sugar is mostly smaller than one poor in sugar. The likely roots are sorted into small heaps in autumn. In spring any beets that are damaged are put aside and the others sorted according to weight and sugar content. A cylindrical piece is bored out from each root and tested for

sugar. The beets which are approved are planted to produce seed, which is gathered when ripe in August and September. This seed is not sold, but a small part is used to grow mother beets for further selection tests. The bulk is sown close, and next year produces the seed used for sale. Thus it takes five years to obtain a select crop of beets from the original mother beet; 20,000 best mother beets are sufficient to plant a hectare (2·47 acres), and from them are obtained 40 to 60 hundredweight of the best seed, and this gives the following year 150 to 250 acres of the best (Elite) beets, or from 5 to 7 millions of plants. From these there are finally chosen the 1½ million seed-bearers, which furnish the planting of 250 acres and the seed for sale and for the perpetuation of the breed. The seed is cleaned as usual, and regulations are made for the trade as to moisture, foreign matters, and germinating quality which it must satisfy. The seed is not purchased indiscriminately, but each factory has its own fancy for the particular variety supposed to suit the soil, climate, and other conditions.

**Composition.**—The cellular tissue of the beetroot is permeated by vascular bundles, the zones of which are allied to the production of the leaves. In the cells is the juice containing sugar, salts, and other organic matters. The insoluble tissue forms 4 to 6 per cent., and the juice 94 to 96 per cent., 75 to 80 of which is water. The sugar content is 14 to 16 per cent., but in some cases, in carefully nursed beets, has been found as high as 22 per cent. The non-sugars, including salts and organic matter, amount to about 2·5 per cent. The inorganic constituents are principally potassium, next sodium, calcium, phosphoric, and sulphuric acids, chlorine and silica. The organic matters include oxalic and other organic acids, invert sugar, raffinose, albumen, pectic substances, gums, amino-acids, betain, colouring and aromatic substances. The qualities of a good beetroot are thus summarized by Saillard: "A rich beet offers the following advantages: it bears more leaves per acre, it contains in its root less mineral matter per 100 of sugar, and its juice is purer. This makes it less

exhausting to the soil and gives less molasses. It needs less mineral matter for the entire plant (root and leaves) to elaborate 100 kilogrammes of sugar, and the mineral matters which it draws from the soil pass in great part to the leaves; it has a firmer flesh, it keeps better in silo, it requires less expense for transport per 100 of sugar, it resists dry weather better, and all this does not prevent it from producing as much, or more, sugar per acre than the semi-sugar beetroot. It must be added, however, that it leaves less factory pulp per acre." In addition, the purer juice is less troublesome to deal with in the factory.

**Manufacture.**—The roots, having been properly harvested and delivered to the factory, are first conveyed to washing tanks provided with suitable stirring gear for keeping them in motion and conducting them towards the end where fresh water enters so that any adhering soil, sand, and stones may be completely removed. The beets are next raised by a suitable elevator to the slicing apparatus into which they are allowed to fall. Here the beets are sliced into thin pieces of greater or less length, of such form that when placed in the vessels of the diffusion battery they will not pack so closely together as to prevent the circulation of the diffusion juice. These slices, called cossettes or chips, are filled into the vessels of the diffusion battery, and there submitted to systematic exhaustion, the diffusion. The exhausted chips are passed through a press to remove a portion of the water, and are then used either fresh, or, after being dried, as cattle food. The diffusion juice is led to the carbonatation or saturation tanks, where it is treated with from 2 to 3 per cent. of its weight of lime, and afterwards with carbonic acid, until nearly all the lime is precipitated (the first saturation). The still alkaline juice is now passed through filter presses by which the precipitated carbonate of lime is removed. The juice next passes to a second set of carbonatation tanks to undergo a similar treatment with lime and carbonic acid, only that a smaller quantity of lime is now used as compared with the first (second saturation). The scums are again removed by filter presses, and the clear yellow juice may be

treated with sulphurous acid (sulphuring) and once again filtered. The purified juice is then concentrated in a multiple effect vacuum apparatus to a syrup. The syrup is sometimes again submitted to saturation with carbonic or sulphurous acid, filtered, and then passed to the vacuum pan to be boiled to grain. The mixture of sugar crystals and syrup, called massecuite, thus obtained, and containing 6 to 10 per cent. of water, is run out into large vessels provided with stirring gear, and kept stirred to continue the crystallization on the sugar grains already present—crystallization in motion, as this is called. The cooled mass is spun in centrifugal machines. The raw sugar crystals remain in the centrifugal, from which they are taken out, sieved, and put into sacks, while the syrup which flows off is treated for second product sugar by boiling to string proof, that is, to a dense syrup which only slowly crystallizes when allowed to cool in tanks; or it is boiled to grain and crystallized in motion to give low sugar and molasses. The molasses is either used for making cattle food, treated chemically to extract the sugar by a desugarizing process, or sent to the distillery to be fermented for the production of spirit.

**Brief History of the Foundation of the Beetroot Sugar Manufacture.**—The beetroot was not considered as having an industrial value, and was cultivated only for table use or for cattle food until 1747, when Marggraf, a Berlin druggist, examined a number of plants other than sugar cane and found the beet to be richest in sugar amongst these. By cutting the root into thin slices, drying these carefully, and reducing them to powder, he was able to extract 5 per cent. of sugar by means of highly rectified spirit from the Silesian beet. He fully realized the wide-reaching importance of his discovery, and advised farmers to extract beetroot pulp with water, clarify the solution with albumen or blood, and evaporate the filtered solution to crystallize out the sugar. His proposal was unheeded until his former pupil, Achard, in 1786, repeated the research on roots he had specially cultivated, and finally succeeded in extracting sugar from the beet on a comparatively large scale. In 1799 he presented a

sample of his sugar with a description of the method of operation to the Institute of France, by whom a commission was appointed to examine the question. The report was not favourable at that time; but when the continental blockade ordered by Napoleon closed the harbours of Europe to all English commodities, it caused an immense rise in the price of sugar and led to the establishment of beet-sugar manufacture in France. Under the influence of Chaptal 80,000 acres (32,000 hectares) of land were by Royal decree set aside for beet cultivation and substantial premiums awarded for the promoters of the industry. Figuier at this period discovered the decolorizing power of bone charcoal, which was used at first in the form of powder. On the fall of Napoleon, in 1814, and the cease of the blockade, the price of sugar fell so that almost all of the factories closed due to the crisis. Those remaining at work were under Chaptal's direction, and their aim was to continually perfect the methods of working: the introduction of char in granular form, boiling in vacuum, the general use of steam and use of many factories as model institutes for instruction, so that in 1828, 103 factories were at work with an output of about 5000 tons. This development and its favourable results with the experience obtained was not without effect on Germany and Austria, where the industry awoke to new life, assisted in no small degree by the fact that it was not shackled by any duty, a measure which soon paid handsomely, when the improved technique and increasing yield enabled the young industry to become strong enough to withstand competition.

**Early Methods of Juice Extraction. Pressing.**—The various methods of extracting the juice from the beetroot which have been successively employed are, briefly, rasping and pressing, centrifugal treatment of the rasped pulp, continuous presses, cold and hot maceration and diffusion. In the earliest of these methods used in France the beets were reduced to a fine pulp by special rasping machines, and this was pressed; originally wooden presses were used, then screw presses, finally succeeded by hydraulic presses. The

pulp was wrapped in strong but coarse woollen cloths, and placed in layers in the press with a metal sheet between, and exposed to high pressure. The expenditure of manual labour in filling and emptying the presses and washing the cloths soon led to the use of roller presses.

**Cold and Hot Maceration Processes.**—The method of pressing extracted only 70 to 80 out of the 96 to 97 per cent. of juice present in the beet. The process of rasps and presses was the one chiefly used in Germany until 1870, and in France until 1876. The introduction of the maceration process or soaking out of the juice from the rasped pulp, marks an important improvement. The levigator constructed by Pelletan in 1836, adopted in several French factories, consisted of an inclined, semi-cylindrical trough in which the pulp was raised by a worm against a descending current of cold water. The water became gradually enriched with sugar, and issued as juice, only slightly more dilute than that obtained by pressing, while the pulp, on arriving at the top, had given up its sugar. The fault of the process lay in the difficulty of obtaining juice free from fibre, which caused trouble in defecation. The cold maceration process of Schutzenbach overcame this difficulty. A number of vessels (usually twelve) arranged in line, mostly in cascade form, are filled with pulp resting on the false bottom. Water is run on the pulp in the highest vessel until full, and the dilute juice obtained is drawn from below the sieve on to the next, and so on, until the pulp in the first vessel is exhausted. Water is then started on the second vessel, and the first (the highest) is emptied. As each in turn becomes the starting-point in the series, the partial juice is pumped from the lowest vessel to the highest, and the dense juice from the one most recently filled is sent to the defecators. Each vessel is provided with stirring gear to mix the water and pulp while maceration proceeds. This process also required the most scrupulous cleanliness to secure juice that could be easily worked. After each vessel was emptied it was thoroughly washed, especially the part below the false bottom which afforded a suitable lurking-place for germs of the

dreaded "frog-spawn" (*Leuconostoc mesenterioides*). This organism develops at a rapid rate, producing the dextran fermentation. The earlier filter presses, too, were often covered with a growth of this undesirable bacterium.

From time to time various combinations of methods were used: pressing the raspings followed by maceration of the partially exhausted pulp, as also removing the greater part of the juice with a centrifugal machine, and the remainder in the same way after macerating in water. In spite of some advantages the latter method was abandoned because of the danger of bursting the centrifugal owing to irregular running from the uneven way the pulp loaded into the machine.

As early as 1830 maceration with hot water was introduced by de Dombasle, who held that the life of the cell must be destroyed by a sufficiently high temperature (60° to 100° C.) to enable the juice to pass out, and also that the maceration must be prolonged for a sufficient time for the extraction to be complete. The process was capable of extracting 90 per cent. of the juice. The beetroots were cut by a machine into slices about 6 millimetres in thickness and filled into a battery of six vessels, provided with false bottom and heating coil. The duration of contact in each vessel was half an hour, and the juice drawn off from each vessel was run into the next following. De Beaujeu, in 1833, installed a similar plant in which the juice passed by gravity from one vessel to the next, and the communicating pipe was utilized for heating the juice. It was further suggested to heat the fresh slices by steam before treating them with juice. The process of de Dombasle was adopted in 1847 by F. Robert, at Seelowitz, in Moravia, but owing to the difficulty of defecating the maceration juice it was mixed with juice obtained by pressing, the two processes being worked simultaneously.

In 1837 Schutzenbach, instead of using fresh raspings from which to extract the juice, proposed to dry slices of beetroot to preserve them. These could afterwards be softened in water to which milk of lime was added, and

extracted in closed vessels with water at 88° C. In this way he expected to make the factory independent of the season and able to work continuously throughout the year. The principal obstacle lay in the difficulty of drying the slices uniformly and without destruction of sugar by overheating, and the hope of securing a more concentrated juice was not realized. This process was worked at Seelowitz along with other methods. At this period J. Robert entered his father's factory, after having made a study of the morphology and physiology of plants as well as of the phenomena of osmosis, diffusion, and dialysis investigated by Dutrochet, Dubrunfaut, and Graham, and had come to the conclusion that since the beetroot cells were able to obtain their nutriment from outside by a purely osmotic process, it should be possible, under suitable conditions, for them to yield their soluble contents, principally sugar, in a similar manner without the necessity of tearing or bursting the cells, as is done when the roots are rasped or when overheated in maceration.

**The Diffusion Process.**—For diffusion the beetroots are cut into thin strips, one millimetre in thickness, and systematically extracted in five or six closed vessels similar to macerators. When the first vessel is being filled with the fresh chips, water heated to 87° C. in an open heater is run in. The diffusion in each vessel lasts half an hour, and the average temperature of the battery is 30° to 50° C.

Robert worked his patented diffusion process for a week at the end of the campaign, 1864–1865, and the following season the factory at Seelowitz used this method exclusively. The results were so satisfactory that in 1867 the diffusion process was used in 27 factories, and in 1885, out of 408 factories in Germany no less than 402 worked it. Diffusion made quick process also in Austria and Russia, but in France was not introduced owing to the opinion that it was unsuitable for the poorer quality of beets grown there. Besides this the sugar factories had neither chemist nor laboratory, and there was some hesitation in introducing a process requiring some knowledge of science. Quarez, however,

started diffusion in 1876, and obtained a better quality of juice and 1 per cent. more sugar in this way than by pressing. It was then objected that the exhausted pulp was injurious to stock, and only in 1884, when the duty was charged on beetroots, the process was definitely adopted.

**Theory of the Diffusion Process.**—The rapidity with which the diffusion process replaced the older methods was a proof of its superiority due to the purer juice it furnished. The torn cells of rasped pulp yielded the whole of the contents of the cells by pressing and maceration of the pulp was simply a lixiviation of this impure juice. The same result followed when the cells were burst by too high a temperature in hot maceration, and prolonged contact with hot water also brought into solution pectous substances and increased the viscosity of the juice. The aim of diffusion is to obtain as much sugar as possible from the beet without at the same time allowing too much non-sugars to enter the juice. As diffusion is a slow process, it is requisite that the roots be cut into suitably thin slices, and these should be exposed to the action of diffusion as long as necessary for equilibrium to be attained between the juice left in the cell and the extract, this diffused juice is then to be replaced by weaker juice, and the operation repeated until the slices are sufficiently exhausted of sugar. There is a limit to the thinness of the slices since the cut surface lays open cells, the contents of which mingling with the diffused juice lower its purity, and the cleaner the cut the less will be the number of cells thus opened; it is thus advisable to work with well-sharpened knives; when these become blunt the cells are torn. Further, diffusion only occurs when the protoplasm is killed or modified so that it can no longer offer an obstacle to the passage of the juice, hence the necessity for the higher temperature in the first diffusion vessel. Apart from this a rise in temperature accelerates osmosis, but the heating must be kept within limits, so as not to injure the quality of the juice. A microscopical examination of an exhausted slice after diffusion shows the cells to be intact and the

intercellular pectose, which only swells in boiling water, is seen to be still compact.

**Extraction of the Juice.**—To prepare the beetroots for the extraction of the juice they are first conveyed to the washing-machines to be freed from adhering earth and stones, then drained, weighed, and cut into chips ready for filling into the diffusers. The beets are now usually conveyed from the store sheds by flumes which are cement gutters about 2 feet deep, 15 inches wide, and rounded at the bottom. These gutters run the length of the sheds and are covered with boards. Water is turned on, and the board at one end is lifted and the beets allowed to fall in and be carried along by the current. As the work proceeds one board after another is lifted until the store is emptied. At the end of the run the beets fall into a drum of perforated metal, of large diameter and open at one side; by this wheel they are lifted and dropped into a shoot leading to the washing-machine. Sometimes an Archimedean screw is used to raise the beets from a well into the washer.

**Washing the Beets.**—The washing-machine consists of a wrought-iron trough with a rounded false bottom perforated by slits through which dirty water may pass into the collecting space below. The beets are tossed about and moved along by a number of arms arranged spirally on a revolving horizontal axis, while a constant stream of water is run through and kept up to the level of the axis. The beets then reach a second compartment of the washer in which stones, etc., fall to the bottom, while the floating beets are lifted by scoop-like arms and thrown out to the elevator, which raises them to an upper story. Here they are drained and weighed.

**Beet Slicing Machines.**—The cutting machine consists of a horizontal disc 4 to 6 feet in diameter on a vertical axis, and making 80 to 100 revolutions per minute. In the disc are a series of 8 to 14 apertures, into which fit the knife-holders on which the knives are screwed. These travel in an annular path to which the beets are confined by baffles in the hopper, surmounting the casing around the disc. A bell

covers the central part of the disc which does not work. Cross-pieces a few millimetres above the disc prevent the beets from rolling and being carried round with the revolving disc so as to ensure their being cut. The hopper is about 6 feet high and wide enough so that the beets may fall freely and exert sufficient pressure, otherwise good chips would not be obtained.

In another form of machine the knives are arranged inside a horizontal cylindrical drum with the cutting edge pointing inwards. The chips pass through the apertures in the knife-holders. The beets are kept in position by a curved prolongation of the side of the feed hopper.

Each knife-holder contains a knife with a set of blades similar to the blade of a carpenter's plane, but formed so as to cut chips of the sectional form desired. One of the most approved forms gives chips the section of which, being similar to the tiles on the ridge of a roof, is called the ridge tile. This form of chip presents a large surface, and the chips lie loosely packed in the diffuser so that the circulation of the juice proceeds freely. Opposite the cutting edge of the knife is the counter blade, which can be raised or lowered and fixed in position by a screw. Its set determines the thickness of the cut chips. The far side of this plate has a number of apertures to allow of the passage of any small stones which have not been removed in the washing. These frequently blunt or break the knives and cause a stoppage of the work. The apex of one knife follows the other in the same circle, and the width of the strip—or from apex to apex —varies from 4·5 to 7 millimetres. The size of knife used is chosen to suit the flesh of the beets being worked, the larger ones are used when the beets cut badly. A disc 5 feet in diameter with twelve sets of knives will cut 300 tons of roots per day. The chips are conveyed to the diffusers by a travelling belt or otherwise.

**Working of the Diffusion Battery.**—The ten or twelve diffusion vessels worked in connection constitute a diffusion battery. Formerly these were sometimes preferred arranged in a circle, but they are now placed in a single or double row.

Each diffuser is a wrought-iron cylindrical vessel, about 5 feet in diameter, and of a capacity of 1750 to 2500 gallons. They taper conically above and below to where the cover closes the manhole for filling, and the door below closes that for emptying the exhausted chips. Each diffuser is connected with a heater and with valves in communication with water and juice conduits. The conical parts are furnished with perforated sheet to act as a sieve, and prevent the chips from passing away and blocking the pipes and valves. The last diffuser (No. 1 suppose) containing the chips most nearly exhausted of juice is supplied with water under pressure from a tank 30 feet above. The dilute juice flowing from the bottom of this diffuser passes through the heater and enters the next diffuser at the top, and follows a similar course through the whole of the diffusers at work, becoming enriched on its passage until it issues, still under pressure, from the first diffusion vessel (No. 10) containing the freshest chips, to enter the measuring tanks as raw diffusion juice through the juice pipe. The amount drawn off is thus known precisely and is varied from 100 to 110 litres, or even more, per 100 kilos of beetroot to suit the conditions. When the chips lie loosely packed there is a greater proportion of juice present to the chips and more must be drawn off to get the same exhaustion. When a diffuser (No. 11) has been filled with fresh chips the course of the circulation is altered to mix them with the juice—mashing as this is called. If the juice entered at the top the air between the chips would become entangled and prevent proper circulation. The valve to the measuring tank is closed and the valve to the heater following No. 10 is opened so that the juice from No. 10 passes along the juice pipe down through the heater and enters diffuser No. 11 below, chasing the air before it. As soon as juice issues from the air cock in the cover these valves are closed and the normal course of the circulation proceeds, No. 11 forming now a working part of the battery. Meanwhile No. 1 is cut out of the circuit and emptied and water is run on No. 2. In order that the juice in all the vessels should

have the temperature considered most favourable, the steam on the heaters is kept full on, but as the chips are filled at about 20° C. the temperature of 75°–80° C. is only reached in the second to the fourth diffuser from the head of the battery; from this point the temperature falls to the last vessel, where cold water enters. In some cases the battery is worked hotter throughout with a quicker exhaustion and a smaller number of diffusers, the chips are heated by a jet of steam underneath the knife plate in the cutting machine and are then hotter when filled, and warm water (40° C.) is used instead of cold. The exhausted chips then, however, require to be cooled before they can be pressed and stored, otherwise they would rapidly deteriorate. Too high a temperature, 85°–90° C., leads to a disturbance of the circulation, the chips are said to be scalded; they become soft and lie close together and against the sieve. The ordinary methods of working differ widely in different factories, as regards the temperature, duration and density of juice and according to the character of the beetroots and of the chips. Diffusion includes lixiviation of broken cells, dialysis and solution of otherwise insoluble matters. The latter is injurious, but is favoured by the same circumstances that help diffusion. Complete exhaustion of the chips is favoured by heat, by thin juice and fine chips, and is more complete if slow, but the juice is purer when diffusion proceeds quickly. When the chips are irregular in thickness a solution of insoluble matters proceeds from the thinner ones while the thicker are exhausting. When frozen or decayed beets are worked, more sugar must be left, otherwise the circulation is stopped if the temperature is raised to help the exhaustion.

Modifications of the ordinary diffusion have been made. The process of Naudet, by bringing the freshly mashed diffuser to the maximum temperature the chips can bear, obtains a quicker extraction and more concentrated juice while the chips are kept quite a short time in contact with hot juice. The juice from the second diffuser enters the first, which is just filled, below and leaves at the top, and

then passes through a heater and is again pumped in below, and this is continued until the juice issues at 75° C. Then the first vessel is put into the usual circuit and the extraction proceeds more quickly, so that only 4 to 6 diffusers are required instead of 8 or 10, and the exhausted chips can still be pressed easily. Perhaps the most successful of the processes of continuous diffusion is that of Hyross Rak. Each battery consists of six upright diffusion vessels about 18 feet in height in which diffusion takes place under a pressure of 1 to 2 atmospheres of water; the chips, however, do not remain in the same diffuser during the whole process but pass from one vessel to the next. This uninterrupted progression of the chips is brought about by a spiral blade in each diffuser and pallets in the transition connecting chambers. Each of the diffusers is somewhat conical in form and fitted with a sheet of perforated metal in the diffusion space, forming a double wall with the exterior. Beyond the sieving zone of each diffuser is a construction in which the chips become compressed and form a wad and prevent the juice of one diffuser from mixing with that of the next. Condense water at about 60° C. from the triple effect is pumped at 1 or 2 atmospheres' pressure into the almost exhausted chips entering the last diffuser. It permeates these and drives the juice separated in the sieving zone through the ascension pipe into the previous diffuser, and so on to the third vessel. The juice from this diffuser is drawn off continuously to serve as mashing juice without heating for the mashing zone of the first diffuser. After mashing, the juice enters the circulating tank, from which it is drawn by a pump and forced through a heater to raise its temperature to 85° or 90° C. The heated juice is supplied to the first diffuser at three different levels in order to allow the juice to mix more readily with the fresh chips and to render the heating more regular. The first diffuser is of 550 gallons capacity, the others 400 gallons, and the battery is capable of dealing with 400 tons of beets per day. The last diffuser acts as a press to the exhausted chips, which leave the vessel in a more or less pressed condition. There

is no residual press water to be dealt with, as this is automatically returned, and the loss of dry solids in this water is thus avoided. The scalding process of Steffen extracts 75 to 80 per cent. of the sugar contained in the beetroot, the remainder is left in the pressed pulp, which is dried to give a dry sugary pulp.

**Exhausted Pulp.**—The spent chips emptied from the diffusers fall into an inclined gutter, and are then raised either by an Archimedean screw or by an elevator with perforated buckets for pressing, to free them, as far as possible, from adhering water. As dropped from the battery they contain 94 to 95 per cent. of water, and when raised represent 80 to 100 per cent. of the original beets. They are pressed so as to contain 10 to 15 per cent. of dry solids before delivery to the farmers, who generally require an agreement to receive 10 cwt. of pulp, at a stipulated price, for each ton of beets delivered. The pulp and sweet water are acid and contain lactic and butyric ferments in a favourable medium and at a suitable temperature for development, so that the gutters require to be washed frequently with milk of lime, and when empty, with solution of bleaching powder.

**Pressing and Drying the Pulp.**—It is more difficult to remove the water from the spent chips by pressing when they have been exposed to too high a temperature during diffusion, or the exhaustion carried too far, or if they are too acid, and the character of the beets also is of influence. The chips are passed through one of the forms of Kluseman or Berggren mechanical press. This consists of a cylinder of perforated metal, in which a revolving hollow cone is arranged, also perforated and fitted with a series of projecting arms arranged in a spiral. The wet chips fed in above are moved downwards by the arms to the narrowing space below and pressed, the water is thus pressed out, and flows through the sieve walls, and is collected, while the pressed pulp falls out by a narrow annular exit. The moisture is thus reduced from 94 to about 90 per cent., or in some of the more effective forms to 85 per cent. The

disposal of the water offers a troublesome problem owing to its containing suspended matters and ferments. The pressed pulp forms a useful fodder and is used either fresh or after being stored in silo, where it alters by souring, and loses weight and nutrient matter amounting to 20 or 40 per cent. of the fresh residue. This loss has led to methods of drying the pulp either by the waste furnace gases or by steam. In drying ovens the chips are moved forward by stirring arms over several stages in the same direction as the flue gases, or they are passed through a revolving drum heated in the same way. Steam drying is effected in heated trays over which the chips are moved by scoops attached to heating pipes. The latter method, although more expensive, gives a better fodder. The moisture by drying is reduced to about 10 per cent., and the dried chips then represent about 6 per cent. of the weight of the original beets. The lighter coloured chips produced by steam drying sell at a higher price than those of a darker hue, which often contain charred portions and are less digestible. The dried chips keep well and, not being burdened with unnecessary water, are suitable for transport.

**Molasses Fodder.**—The whole of the molasses made in the factory may be turned into fodder by running it in a thin stream on to the pressed chips before they are dried, and then passing the mixture through the oven. The feeding value of the carbohydrates, as well as the nitrogenous constituents, is thus utilized.

### Purification of the Juice

The various constituents of the beet juice do not diffuse equally, hence the proportion of solids in diffusion juice differs from that originally existing in the cells, and also from that in the sweet waters or retained in the spent chips. It is found that in a juice containing 12 to 14 per cent. of sugar about 97 per cent. of sugar, 65 of the salts, 25 of the albuminoids, and 90 per cent. of other nitrogenous matters have been extracted, so that the diffusion juice is purer. Besides the soluble matters diffusion juice always contains a

considerable amount of fibre and cell fragments. In order to free it as far as possible from this pulp the juice, on its way from the battery to the measuring tanks, is passed through one or more sieves in a closed vessel, the depulper. The raw juice thus obtained still contains fine particles of fibre and cells which have passed through the pulp catcher, and micro-organisms and ferments as well as matters precipitable by heat. These if left in would give rise to acidification and inversion of sugar, and the raw juice is a turbid, grey, opaque, unfilterable liquid which, if boiled to sugar, would give a product of very unattractive appearance and produce much molasses. By the defecating action of lime and heat a considerable part of the impurities is precipitated and the juice is converted into a bright pale yellow filterable liquid, but alkaline from the action of the lime used. The excess of lime is removed by carbonic acid in the process of carbonatation.

**Defecation.**—The purification of beet juice by treatment with lime was adopted from the method long previously in use for cane juice. As early as the seventeenth century wood ashes, lime, or alum had been used for this purpose in America. When the cane juice was heated to coagulate the albuminoids its natural acidity would cause inversion of the sugar unless it was previously neutralized, and lime was found to be better than wood ashes for this purpose, since it produced precipitates with oxalic, malic, and citric acids present which were insoluble, especially in hot juice. Achard, however, in his early attempts to manufacture sugar from beet juice used dilute sulphuric acid to effect defecation. The risk of inversion from excess of acidity soon led to the use of lime as a safer reagent. The quantity used then was very small, being only about 0·5 per cent. instead of the 3·5 per cent. used at present. This was added in the form of milk of lime to the juice at 70° C., and the temperature was slowly raised until the impurities separated as a scum and formed a head on the surface, from which the clear juice below was drawn off. Frequently this amount of lime was found to be insufficient to clarify the juice,

especially if spoilt beets were used, and more lime had to be added to cause the precipitate to separate, so that the juice when drawn off was alkaline, containing sucrate of lime and therefore difficult to boil and crystallize. To remove the excess of alkalinity dilute sulphuric acid was added, until the juice was only faintly alkaline to litmus. Kühlmann of Lille, in 1833, proposed the use of carbonic acid to neutralize and remove lime, and Rousseau, in 1848, adopted the method, preparing the gas by passing air over a coke fire, and passing it through the clear defecated juice until it was nearly completely saturated. The correct point was determined by taking a sample in a spoon and observing if the precipitate of calcium carbonate separated readily; subsequently turmeric was used to decide the proper degree of neutralization. The slight residual alkalinity was removed by passing the juice over animal charcoal. The defecating action of lime on the juice is partly chemical and partly mechanical. The solubility of lime in water is 0·12 gramme per 100 c.c., while a 10 per cent. sugar solution dissolves 1·5 grammes, so that the whole of the reagent used would be present as monosucrate or bisucrate of lime. This begins to form insoluble trisucrate of lime at 80° C., and the precipitation is complete at 100° C. The formation of this insoluble sucrate must be avoided, otherwise there would be a considerable loss of sugar in the precipitate which would form along with the other impurities. The lime neutralizes the acidity of the juice, forms insoluble precipitates with some of the organic and inorganic salts present, and also with albuminoid and pectin substances; these precipitates carry down with them the mechanically suspended impurities; altogether about one-third of the non-sugars are removed. The lime also acts on invert sugar and amino- compounds, converting them into other soluble compounds less harmful in the succeeding operations.

**Single and Double Carbonatation.**—While occasionally the juice that has been merely defecated with lime is decanted or filtered and carbonated almost to neutrality before being used in the factory (single carbonatation), it is now the

general practice to carbonate the turbid juice without previous separation of the precipitate, as was done by Possoz in 1859, and by Jelinek in 1864.

By this means it is possible to use a larger quantity of lime for defecation and obtain a better purification. From 2 to 3·5 per cent. of lime is used. Too small a quantity does not give scums that filter easily after carbonatation; larger amounts are required when decayed or frost-bitten beets are used. Excessive amounts are not only unnecessarily expensive, but the clearer juices obtained are not in proportion to the extra work entailed, and the larger bulk of scums leads to loss of sugar.

If the lime is added in lumps a separate defecating pan is required, provided with stirring gear, heating coils, and a grid on which the lime rests while it slakes. When the pan is sufficiently full the juice is heated to about 80° C., a weighed quantity of lime is added, and the stirring gear started. The lime slakes in a few minutes, and is distributed throughout the juice, which is then run off into the saturator. If milk of lime is used, it is usually made to 20° Be. for convenience of measuring, and the defecation does not need a separate vessel, but is carried out in the carbonatation tank. Defecation may be made continuous by running in raw juice below, adding milk of lime at a regular rate, and having the tank large enough to allow of the overflow juice being sufficiently limed.

The first carbonatation is then commenced. At the start the major portion of the lime is in suspension, the filtered juice shows an alkalinity of 0·3 of CaO per 100 c.c., but as the carbonic acid gas is passed in, and the dissolved lime is precipitated as carbonate, fresh lime dissolves, and the alkalinity is quickly reduced to a certain point where it remains almost stationary. This point corresponds to the formation of the viscous hydro-sucro-carbonate of lime of Boivin and Loiseau, called "cheese" by the workmen. This gelatinous compound, $C_{12}H_{22}O_{11} . 3CaO.Ca(OH)_2$ contains sugar in the insoluble condition, but is gradually decomposed by heat and carbonic acid, and the alkalinity again falls to

about 0·1 gramme of CaO per 100 c.c. when carbonatation is stopped. A sample withdrawn from the tank ought then to show a granular precipitate, which separates and settles rapidly and indicates that filtration will proceed easily. Any of the gelatinous compound left undecomposed would result both in a loss of sugar and difficult filtration. Carbonatation may be made continuous by running in the juice at the bottom of one tank along with carbonic acid; these mix as the liquid rises, and then overflow to the bottom of a second tank where the partly carbonated juice meets a second supply of gas. Less of the gelatinous precipitate is formed, and with good lime for defecation the method works well, but in other cases it often gives rise to irregularities. After carbonatation the juice is run into a collecting and mixing tank, and is then passed to the filter presses, sometimes heated on its way by heaters to facilitate filtration. The point at which the first carbonatation is interrupted, indicated by the rapid settling of the precipitate and the consequent easy filtration of the juice, is readily determined by an experienced workman, but to avoid any chance of error, and to keep the operation under strict chemical control, it is preferable to supplement the test by titrating a filtered sample of the juice to determine its alkalinity to phenolphthalein. The actual percentage of CaO allowed in the carbonated juice depends on the character of the beets worked during the season, and is usually fixed at about 0·10 per cent. The juice therefore still contains a considerable amount of lime in solution. Had the gas been passed through to complete neutrality before removing the precipitate, a portion of the precipitated impurities, which only remain insoluble in presence of lime, would have been redissolved and the juice have acquired a darker colour. Such juice is said to be over-saturated.

The clear filtered juice from the first carbonatation is heated to about 95° C., and again saturated with carbonic acid. In many factories 0·1 to 0·2 per cent. of lime is added as milk of lime to the hot juice before this second carbonatation in order to obtain a further purification.

Saturation is continued until the juice on titration shows an alkalinity of 0·04 to 0·05. There is then no free lime or sucrate left in solution, the residual alkalinity being due to alkalis, ammonia, and organic bases. The saturation pans are round or rectangular closed vessels of wrought iron, made as high as possible to allow for frothing. The height is from 16 to 18 feet, and the other dimensions are arranged to suit the volume of juice treated, which occupies a little over a third of the height. The juice is admitted below. There is also a wide pipe for introducing the carbonic acid, which is distributed below by a perforated pipe or coil, the perforations being directed downwards to avoid obstruction due to incrustation by the lime. The more completely the gas is sub-divided, and the smaller the bubbles, the better is the carbonic acid absorbed and the quicker the saturation. The unused gases escape into the open air through a pipe at the top, passing through the roof. A smaller pipe introduces steam used both for heating, by a coil below, and for breaking the froth, by jets in the upper part of the tank.

**Sulphiting.**—Sulphurous acid combines with lime to form calcium sulphite which, like the carbonate, is insoluble in alkaline solutions. It likewise decolorizes the juice, while carbonic acid always leaves the fully saturated juice darker. The second carbonatation juice, after being filter-pressed, is in many factories treated with sulphurous acid gas or a mixture of this and carbonic acid, but this treatment is generally considered to be superfluous, since the decolorization produced by sulphurous acid only occurs when neutralization is carried very far and there is then a risk of the faintly alkaline juice becoming acid during concentration, when inversion would take place and loss of sugar ensue. It is preferable to apply the sulphurous acid to the partially evaporated or thick juice. The only advantage it seems to offer is that it may decompose calcium salts of organic acids that are not attacked by carbonic acid, and it is held by some that sulphited juices boil more freely.

**Filtration.**—The earliest methods of filtering off carbonatation scums was by means of cloths stretched on frames,

but the process was slow, and there was a great loss of sugar from the large quantity of juice retained in the scums. Bag filters were then used, from which a part of the juice could be pressed out. A great improvement was made by the introduction of the filter press originally devised by Needham, in 1828, for removing water from clay puddle in the porcelain manufacture. A press of this kind, shown at the International Exhibition in 1862, led to its adoption at the Seelowitz factory of J. Robert, where in the form modified to suit the purpose it contributed towards the success of the diffusion process. The first and second carbonatation juices are pumped through filter presses at a pressure which is allowed to rise gradually to 40 or 45 lbs. per square inch. By this means firm spongy cakes are obtained, which fill the chambers of the press, and which are easily washed, and separate from the cloths when the press is opened. Too sudden a pressure at the start is liable to choke the pores of the cloth and impede filtration. The presses of the second carbonatation naturally filter more easily and run a longer time than those of the first carbonatation which give a much more impure deposit. The juice is always submitted to a third and final filtration before it is sent to the evaporators. Filter presses may be used for this purpose, but the juice is then not pumped but run in by gravity from a tank, giving only a few feet head of pressure. In the tank the juice is kept boiling hot to ensure the complete deposition of carbonate of lime, the last portions of which separate slowly. Instead of filter presses for the third filtration some form of mechanical filter is used with a large filtering surface. These are formed of bags stretched over open frames or gauze and suspended in the filter tank. Filtration proceeds from the outside to the inside of the bag, the filtered juice issuing through a pipe over which the mouth of the bag is tightly secured and which is open below to the inside of bag. These filters are worked under a slight pressure only, as too great a force would drive the fine particles of precipitate through the pores of the cloth. Various forms of sand filter are also used as mechanical filters and prove effective.

**Treatment of Scums.**—The press cakes contain about 50 per cent. or more of juice and therefore 6 or 7 per cent. of sugar, most of which must be recovered. By washing the cakes with water at 30 to 40 lbs. pressure the sugar may be completely removed, but to avoid excessive dilution of the juice the sweetening off is stopped earlier, leaving 1 to 2 per cent. of sugar in the washed cake. 1 per cent. of lime will, therefore, give 3·5 to 4 per cent. of washed cake containing 50 per cent. of water. The solids consist, besides the small amount of sugar, of calcium carbonate with the impurities removed from the beet juice and those in the lime used. The organic matters in the cake form about 6 or 7 per cent., the greater part of the albuminoids being removed. Phosphoric and sulphuric acid originally present in the juice are removed as insoluble calcium salts. The washed cake is of value for agricultural purposes since it contains all the phosphoric acid and part of the nitrogen of the beets. When 2·5 per cent. of lime has been used for defecation, 100 of beetroots yield about 12 of washed press cake from the two carbonatations, or 84 of cake to 100 of sugar from beetroots containing 14 per cent. of sugar. Sometimes the cakes obtained are soft and smeary, and do not fill the chambers of the press, in which case filtration and washing are slow and difficult. Attention should then be directed to see that saturation is properly conducted and that the temperature in the diffusion battery is not too high. When the trouble is due to unripe beetroots the only remedy is to change the cloths frequently.

**Lime Kilns.**—The large quantity of lime required for defecation makes it economical for each factory to prepare the lime it requires by calcining limestone in kilns and using the gases produced for carbonatation. The usual kiln is a shaft of wrought iron lined with fire-bricks and tapering slightly above and below, and at least 30 feet in height. It is supported on columns so as to leave a space below where the burnt lime collects and is withdrawn. The opening at the top is fitted with a funnel or bell through which supplies of limestone and fuel (coke or anthracite) are introduced. During work the opening is closed by a cover so that the gases

may be drawn off by a pump through a side pipe ; when the pump is not working the gases ascend by an exhaust pipe and escape into the atmosphere. The limestone is completely burnt to lime in the hottest zone, which is just above the widest part of the kiln where the limestone, heated in its descent by the ascending hot gases, arrives partly calcined in its passage, and as it descends below the hottest zone the glowing lime is cooled by the incoming cold air before it drops underneath the kiln. The lime to be effective in defecation should be used as fresh as possible, and in burning care should be taken that the temperature of the kiln is not allowed to rise too high, otherwise the lime is dead burnt, has the appearance of porcelain and slakes very slowly, and leads to difficulties in carbonatation. This tendency is increased by the presence of silica, alumina and iron, so that limestone selected should be as free as possible from these impurities as well as from magnesia. A good limestone ought to contain at least 97 per cent. of calcium carbonate.

**Carbon Dioxide.**—The gas used for carbonating should be free from noxious constituents and contain at least 20 to 25 per cent. of carbon dioxide, consequently boiler flue gases which contain only 10 to 15 per cent. are unsuitable for the purpose. The carbonic acid produced in the kiln is derived partly from the combustion of the coke fuel and partly from the decomposition of the limestone. Pure limestone burnt with 10 per cent. of pure carbon with air should give theoretically a gas mixture containing 47 per cent. of $CO_2$ by volume. This result is never reached in practice ; because, to avoid the production of carbon monoxide, an excess of air is always used ; it is satisfactory to reach 30 per cent. The quantity of coke fuel should be proportionate to the needs of the kiln ; too little does not give a sufficiently high temperature and unburnt lime results, while the percentage of carbon dioxide is low. Too much coke raises the heat unduly and likewise gives a gas poor in carbon dioxide and possibility of over-burnt lime. The gases leaving the kiln at about 450° C. are too hot to be used directly ; they are, therefore, passed through a washer to

cool them and to free them from flue dust, smoke, soot and tar which have been carried forward owing to the velocity with which they have been drawn off. These impurities would otherwise injure the pump and also contaminate the juice. In the washer the gases entering below meet a cascade of cold water flowing over a series of trays or over a mass of coke, and issue cooled at the top to the pump. The amount of water used must not be excessive, since carbonic acid is fairly soluble in water.

**Sulphur Burner.**—The sulphurous acid used for sulphiting juices and syrups is generally obtained by burning sulphur in an iron vessel supplied with air either by compression or by suction, and fitted with exit pipe lined with hard lead and cooled by a current of cold water in a jacket leading to a washer. Liquefied sulphur dioxide is employed only where the factory is situated so that the question of freight renders this possible. When a compressor is used the air is dried by passing it over a layer of lime or calcium chloride before it reaches the oven. The supply must be under strict control, for if the volume is too great there is danger of extinguishing the flame. Another method of supplying the air is by drawing off the acid gas by means of a Kjorting's jet with steam; this is under easy control, but the air drawn in is direct from the atmosphere and is moist.

## Evaporation

With the final mechanical filtration the purification of the juice is completed, and it should then be quite bright and of a pale yellow colour or, when inferior beets are worked, of a brownish tint. Its sugar content is 10 to 12 per cent. and the total solids as indicated by the Brix hydrometer one degree higher. Its further treatment for the purpose of obtaining the sugar in crystals is carried out in two stages, in the first of which, evaporation, the juice is concentrated to a syrup or thick juice of 50 to 60° Brix, in which no crystals are formed, and in the second, boiling, it is further concentrated to a crystalline mass containing 90 to 95 per cent. of dry solids. The quantity of purified thin

juice is greater than that of the raw diffusion juice owing to dilution by the addition of the sweet water from press washings and the water condensed from open steam used for heating. If 100 kilos of beets give 110 litres of raw diffusion juice the amount of thin juice obtained will reach 125 litres. In order to concentrate this juice at 12° Brix to 60° Brix, 80 per cent. of the water must be evaporated, or an amount equal to the weight of the original beetroots. In a medium-sized factory working 700 tons of beets per day it is, therefore, necessary to evaporate over 150,000 gallons of water, and the importance of efficient plant for dealing economically with this large quantity is so obvious and pressing that efforts at improvement in evaporation have now attained a perfection reached in no other industry. Heating the juice in open pans over a direct fire in the primitive days besides consuming an immense amount of fuel was destructive of sugar owing to contact with excessively hot walls of the vessel. The gases produced by combustion gave up only a small part of their heat and escaped at a very high temperature, and the whole of the latent heat of the vapour formed was lost. The use of steam for heating invented by Watt in 1780, boiling under reduced pressure on the principle of the vacuum pan invented by Howard in 1811, multiple evaporation with vacuum apparatus conceived by Rillieux in 1830, and the various types of film evaporators mark the principal steps in the economical utilization of heat for effecting evaporation. The boiling-point of water or of an aqueous solution is lowered by reducing the pressure, or in other words, increasing the vacuum, under which it boils. Water at atmospheric pressure boils at 100° C. and gives off vapour at the same temperature; under a vacuum corresponding to a pressure of 600 millimetres of mercury water boils at 61·6° C., so that steam at atmospheric pressure, because it is hotter, may serve to boil water under this vacuum. Thus if there are several vessels each under a higher vacuum than the preceding one, direct steam above the atmospheric pressure in the first may boil water in this, and the vapour generated from it boil water in the next and so on. This

applies to sugar solutions with the difference that the boiling-point of the juice is higher the denser the juice. Thus, the boiling-point of juice at 10° Brix is 0·1° higher than that of water, at 20°, 0·3 higher, at 50°, 1·8 higher, and at 60° C., 3·5 degrees higher than that of water, whatever the pressure. Not only must a greater difference in temperature be maintained to ensure boiling, but the greater viscosity of the denser juice retards the transmission of heat and renders the boiling slower; the result, however, is that 1 kilo of direct steam used in the first vessel has been made to evaporate 2, 3 or 4 kilos of water from the juice according to the number of vessels used in the series. This is the principle of multiple evaporation in which the juice partially concentrated in the first vessel of the multiple effect is led into the second where, although denser, it is under a higher vacuum, and the boiling-point is so far lowered as to leave sufficient difference of temperature between it and the vapour from the first vessel for boiling to go on. Similar relations hold between the second and third.

The thick juice pumped from the last body of the multiple effect is no longer clear owing to the presence of a fine, flocculent precipitate formed during concentration, and the colour is now dark yellow or brown. The darker colour as compared with thin juice is partly a consequence of concentration, but also due to the formation of brown colouring matters owing to the action of the high temperature on sugar. The alkalinity has not increased in proportion to the concentration, the decrease being due both to volatilization of ammonia and to the decomposition of nitrogenous non-sugars of alkaline reaction and combination with acids derived from the sugar. The residual alkalinity is still too high for boiling, and the thick juice is therefore sulphited. It leaves the last body at 70° C., and is heated to 100° C., saturated and filtered. Filtration of the thick juice is sometimes difficult, especially if highly concentrated, and for this reason it is often preceded by sulphitation and filtration of the intermediate juice at 30° Brix, and again filtered on leaving the last body of the multiple effect.

**Multiple Effect Evaporators.**—Two forms of multiple effect evaporator are in use, vertical and horizontal, each consisting of 3, 4 or more vessels, and forming a triple, quadruple or quintuple effect. Each body of the vertical form is a tall upright iron cylinder fitted with a heating arrangement, the calandria, consisting of a number of brass or steel tubes, expanded above and below into the tube plates. The juice circulates upwards inside the tubes and descends by a wide pipe in the centre. The space above the tubes is made as high as possible to allow for the spray to settle before the vapour passes on to heat the next body, otherwise a juice collector is placed in the connecting pipe in which by means of baffles the vapour is made to deposit the drops of juice. In the first bodies of the effect the danger of entrainment is slight because the vapour formed is only under a slight vacuum and occupies but a small volume, while the liquid is very fluid, and the velocity of the issuing vapour is not great. But in the next bodies the syrup is more viscous and the vapour occupies four or five times the volume, owing to the higher vacuum, so that when the bubbles burst there is a far greater tendency to entrainment, and a juice collector is indispensable for the last body of the effect. The calandria is provided with a pipe for carrying off the water of condensation from the steam or vapour, and another entering flush with the upper tube plate to carry off incondensible gases and particularly ammonia from the intertubular space of the calandria. This is more particularly necessary in the later bodies of the effect, where the ammonia from the vapour would otherwise corrode the brass tubes. The separate bodies are each provided with thermometer and vacuum gauges, with light glasses and grease cock for introducing oil or grease to keep down froth, and with the necessary valves and connections. The vapour from the last body of the effect after passing through the juice catcher and traversing a surface condenser reaches the air pump which removes the air and incondensible gases and maintains a vacuum of 600 mm. of mercury in the last effect. This vessel acts as a condenser to the

previous vessel, and this to the next, so that a difference of about 20° C. is maintained between each vessel, sufficient to keep up the boiling and concentration of the juice.

The horizontal form of multiple effect consists of elongated pans of somewhat rectangular section, the upper part being semicylindrical and with the heating tubes placed as low down as possible; the steam circulates through the tubes and the juice is outside. The fittings are the same as in the other form, but as there is no great height of vapour space for spray to settle in, each vessel is surmounted by a juice catcher. As the juice becomes more concentrated, more or less precipitate forms, and a portion of this adheres to the tubes as an incrustration, hindering the transmission of heat, so that the tubes require to be frequently cleaned. The incrustation is much more easily removed from the inside of the tubes in the vertical apparatus than from the outside of the tubes in the horizontal form, the tubes being less accessible. For this reason horizontal evaporators are sometimes used for the thin juice and vertical ones for the thicker juice where the deposit is greater. The incrustation consists of carbonate and oxalate of lime with a portion of sulphite when the juice has been sulphited. There is also a certain amount of lime soap derived from the grease used to prevent foaming. The deposit is removed by boiling with a solution of 1 per cent. carbonate of soda and then with 1 per cent. hydrochloric acid.

### Boiling

The filtered thick juice is collected in supply tanks usually provided with floats to indicate the level of the juice, and is drawn off as required into the vacuum boiling pan. Boiling may be regarded as a continuation of evaporation, but differs in both method and aim; while in evaporation the operation is continuous and produces a syrup free from grain; in boiling, on the other hand, the operation is intermittent, each charge being worked up independently, and the object is to cause the sugar to separate in crystals and to form these as uniform in size as possible, so that they may be easily separated from

the mother syrup. It is not possible, however, in one operation to bring the whole of the sugar contained in solution in the juice to crystallization; this can only be done gradually in several steps until there is finally obtained a syrup, molasses, with still a high proportion of sugar, but from which, owing to the quantity of non-sugar present, no further crystallization of sugar is possible.

There are two methods adopted for producing crystals from sugar solutions: (*a*) boiling to grain, in which the juice is so far concentrated in the pan that crystals appear, and these are then made to grow by suitably conducting the boiling, and (*b*) boiling to string proof, when a small sample of the thickened syrup is placed between the thumb and forefinger and these drawn apart forms a thread, adapted for syrups of low quality or for juices arising from a bad quality of beets; in this case the syrup is merely concentrated at a high temperature in the pan, and then run out and brought to crystallization by cooling. The presence of non-sugars influences the crystallization, and when the proportion exceeds 20 parts in 100 of dry solids, boiling to string proof is the only method generally available.

Both methods of boiling depend on the proper regulation of the conditions of saturation and super-saturation. A solution of pure sugar at a given temperature is saturated when it is in equilibrium with crystals of solid sugar, and neither dissolves sugar from the crystals nor deposits sugar upon them in the crystalline form. The solubility of pure sugar in water as determined by Herzfeld is given in the following table, which shows the number of grammes of sugar dissolved by 100 grammes of water at the corresponding temperatures:

| 0° | 10° | 20° | 30° | 40° | 50° | 60° | 70° | 80° | 90° C. |
|---|---|---|---|---|---|---|---|---|---|
| 179 | 191 | 204 | 220 | 238 | 260 | 287 | 320 | 362 | 416 |

Thus, 462 grammes of a saturated solution at 80° C. when cooled to 40 degrees will deposit 362 — 238 = 124 grammes of sugar. When a saturated solution is cooled, however slightly, it becomes super-saturated, so also if some of the water is evaporated while the temperature remains constant,

and the super-saturated solution crystallizes more or less rapidly dependent on the degree of the super-saturation and on the purity of the solution. For the speedy deposition of sugar on crystals already formed only a slight super-saturation is required; if it becomes too great the increased viscosity of the solution impedes circulation, and there is a tendency to the formation of a crop of small crystals, false grain, as they are called.

**The Vacuum Pan.**—Boiling under reduced pressure and, therefore, at a lower temperature, becomes of greater importance with the more concentrated syrup required for crystallization, because the high temperature at which it boils can more readily cause destruction of sugar by caramelization, and especially in the presence of free alkali. Prolonged subjection to heat in the pan is also injurious, and an endeavour is made to complete the boiling in as short a time as possible by increasing the difference in temperature between the juice and the heating steam both by increasing the vacuum and by raising the pressure of steam in the coils. This period, however, is limited by the rate at which crystallization can go on increasing the size of the crystals already formed without the production of a fresh crop.

Small spherical copper vacuum pans are now used only in smaller factories, and are superseded by larger cylindrical pans of cast iron which are more easily constructed and lend themselves more readily to lagging with non-conducting material to prevent loss of heat. These have generally the same form and construction as the bodies of the multiple effect with the modifications essential to allow of the free circulation of the viscous and pasty final product, the massecuite. The outlet valve is large, and there is a cock for steaming out any masses of sugar left behind when the pan is emptied. There are three or more large copper heating coils which can be worked separately or in combination according as the contents of the pan increase in height during the boiling. If the calandria form of heating arrangement is adopted the tubes are made wider and shorter to allow for circulation of the massecuite, and with a much

wider tube in the centre. In the horizontal form of pan the tubes are arranged in rows vertically over one another with wide spaces between.

**Boiling to Grain.**—It is only juice of high purity that can be readily boiled to grain: juices of lower quality are sometimes enriched by melting with them the massecuite fine-grained dark sugars from low products. A quantity of juice is drawn into the pan sufficient to cover the lower coil and concentrated to string proof. By the length of the thread formed when a drop of the syrup is drawn out between the finger and thumb it is described as weak or strong proof. For graining thick juice the proof should be rather light. Small quantities of juice are then drawn in suddenly, and owing to the cooling and agitation which ensue crystals are formed in the syrup. Samples are drawn by the proof stick and spread on a glass plate, and from the appearance it is judged whether a sufficient number of crystals have been formed. If so, a larger quantity of juice is drawn in to lower the super-saturation, and this procedure is repeated as often as the proof shows the need for further supplies. As the grain increases in size the panman watches the samples to see that the surrounding syrup is clear and free from small false grain. The indications of the thermometer and vacuum gauge are also noted for guidance, as also the splashes on the sight glasses. The volume of the juice in the pan increases, and as each coil becomes covered, steam is admitted to it, and boiling is continued until grain of the desired size is reached. For raw sugar a coarse, sharp grain is desirable, but for sugar that is to go into consumption direct the grain should be fine. Large grain is obtained by boiling quietly and drawing in large drinks of juice at long intervals during which the crystals grow; finer grain is produced by more vigorous boiling and frequent small supplies of fresh juice. The end of the boiling is indicated by the condition of the massecuite, which should show a large quantity of crystals and only a small quantity of syrup from which the crystals separate distinctly. The massecuite as dropped from the pan should be quite stiff, but crumbly. The time of boiling

a charge is from 6 to 8 hours, and when syrups from a previous operation are drawn into the pan at the end, the boiling may proceed for another hour or two. Boiling does not always proceed normally. Occasionally a quantity of thick froth forms as soon as the juice begins to boil, and there is danger of the whole mass boiling over. This is to some extent overcome by boiling at a lower vacuum and higher temperature, and by the introduction of grease into the pan. The cause is often to be traced to imperfect filtration, or the use of an insufficient amount of lime and faulty defecation. When bad or decayed beetroots have been worked the juice often boils badly and almost ceases as soon as a certain concentration is reached, the contents of the pan remaining motionless like melted fat. The grain forms only with difficulty and grows slowly, and the transmission of heat into the thick mass is so small that caramelization ensues, and the juice becomes dark brown. Slow and difficult boiling has also been attributed to presence of alkali and to calcium salts of arabic acid. The remedy is either to boil merely to string proof or to neutralize with sulphurous or phosphoric acid.

**Control of the Boiling.**—The conduct of the boiling operation is in the hands of an experienced panman who has served an apprenticeship and has learnt from the appearance of the mass how to regulate the super-saturation and the consequent course of formation and growth of the crystals. Boiling to grain was for long considered an art and beyond the control of science. In recent years, however, several forms of apparatus have been introduced to secure this control, among which may be mentioned the brasmoscope of Curin and the apparatus of Claassen. These are slide rule arrangements by which the temperature of the boiling mass in the pan and the vacuum give the percentage of sugar or degrees Brix of the syrup, and this, in conjunction with the degree Brix of a saturated solution of sugar at the same temperature, shows whether the syrup in the pan is or is not saturated or super-saturated. Moreover, it is possible to express the degree of super-saturation as shown by Claassen. If the ratio of sugar to water in a saturated solution be

denoted by S, and the ratio for the syrup in the pan at the same temperature be $S^1$, then $S^1/S$ represents the degree of super-saturation if $S^1$ is greater than S. For graining thick juice the concentration is carried out until this ratio is 1·2 to 1·3, and after the grain is formed, the super-saturation is reduced to 1·05. With lower-class products the ratio requires to be somewhat higher, and in any case, if records are kept of the course of a well-boiled pan, the operation can be safely repeated with the aid of the indicators, preserving the same ratios at the different stages of boiling, and thus avoiding errors of personal judgment. The apparatus, however, only affords reliable indications provided the contents of the pan actually boil.

### Treatment of the Massecuite

When the massecuite is discharged from the pan it is at a temperature of about 80° C., and the syrup is somewhat strongly super-saturated, so that if the crystals were separated at once the syrup would carry off an undue amount of sugar into the lower product. If the mass is allowed to cool quickly the super-saturation increases so much that a crop of very small crystals is formed which pass off with the syrup from the centrifugal machine, being too fine to be retained. To secure this sugar in the first product it is, therefore, necessary to moderate the rate of cooling and to lessen the super-saturation by the periodical addition of somewhat diluted syrup from a previous operation, or even water, as the temperature falls. By this means the viscosity and super-saturation are kept low enough for the sugar to deposit on the crystals already present, and this result is assisted by keeping the whole mass in slow movement until the temperature has fallen to about 50° C. It is not advisable to cool below this temperature, otherwise the mass would become too stiff and not possess the necessary mobility to enable it to rise in the centrifugal machine and form a uniform layer. The whole contents of the pan are let down into a crystallizer which is either a closed horizontal cylinder or a large open trough provided with stirring arms with a

revolution in one or two minutes. The crystallizers are often jacketed so that they may be heated with steam or hot water, or cooled with water during the 12 to 24 hours of crystallizing and cooling. The cooled massecuite may be drawn off in bogies running on an overhead rail, and each carrying 2 to 5 cwt., sufficient for one centrifugal machine, or it is passed along a gutter with a screw conveyor. From the latter each machine draws its charge.

**Centrifugal Treatment of the First Massecuite.**—As soon as the drum of the centrifugal is set in motion, and while it is getting up speed, the massecuite is run in. The whole mass as it falls is gradually thrown against the wall of the basket and rises to form a uniform layer. The sugar crystals are retained, while as the speed increases the liquid syrup passes off through the holes or slits in the metallic cloth of the basket and strikes against the wall of the outer casing, from which it trickles down to the gutter, and is run off as first product drainings. The centrifugal is run as long as syrup comes away, but it is not possible to remove the whole of the syrup in this way, a thin, strongly adherent layer remaining on the crystals. This may amount to some 10 per cent., dependent on the character of the grain. The raw sugar obtained has a net rendement of 88 to 92 per cent. When sugar is required for direct consumption it is necessary to remove the greater part of the remaining adherent syrup which gives to beetroot sugar its unpleasant taste. This is possible when juice of a high degree of purity has been worked and when the defecation has been carefully carried out. After the first drainings syrup has been removed as far as possible in the centrifugal, the sugar is washed with water, steam or with a saturated solution of pure sugar, which are introduced while the drum of the centrifugal is still in motion. The washing removes most of the remaining syrup and yields a white sugar, but it cannot be regarded as a refined sugar which must have undergone solution and purification. The syrup from this operation is, of course, richer than the green, mother syrup first spun off, and is returned to be boiled with the thick juice.

**Crystallization in Motion of the Second Massecuite.**—The green syrup runnings from the raw sugar were formerly boiled to string proof and the concentrated syrup left at rest in shallow iron tanks holding several tons each. After remaining for some days the whole became a mass of fine crystals and syrup, or a deposit of fair-sized crystals with a layer of syrup above. The cold mass was dug out, passed through a pug mill to make it uniform and mobile, and dried in the centrifugal. The syrup drainings were treated in a similar manner to give a third-product sugar and molasses, but required a few months for crystallization to be complete, and the tank rooms had to be kept warm during all this period to render the rate of cooling slow. The third-product sugar was frequently so dark and sticky as to be unsaleable, and had to be re-melted and worked with the raw juice. More recently the centrifugal syrups from the first product are boiled to grain and the massecuite introduced into crystallizers and kept in motion as already mentioned under first-product treatment. Owing to the accumulation of non-sugars in the syrup the grain is more difficult to form in this case and slow in appearing, but it is always possible to grain the syrups unless they are too impure. After grain is formed it is built up gradually into crystals during a boiling lasting 18 to 24 hours, when the pan is then full. It is an advantage if the pan is provided with stirring gear, the rotating arms of which are arranged above and below the calandria. The massecuite is then let down into the crystallizer and kept stirred for 4 or 5 days, while the temperature is allowed to fall from 90° to about 40° C. Syrup or water is added from time to time as may be required to keep the mother syrup from becoming too highly super-saturated, lest false grain should be formed. At the end of this time the mass may be centrifugalized direct. The syrup runnings constitute molasses. Another and earlier method of maintaining the proper degree of super-saturation was to raise the temperature a few degrees as soon as that of the massecuite had fallen about 10°, and to repeat the warming for each similar fall, by passing steam or hot

water through the jacket of the crystallizer. Crystallization in motion not only gives sugars of better quality and higher yield than were obtained by the older method, but the second product is obtained within a few days after the end of the campaign instead of the months required by crystallization in tanks and the expense of keeping the rooms warm all this time.

**Molasses.**—Factory beetroot molasses has an unpleasant saline taste and disagreeable odour, and for this reason is unfit for human consumption, and the attempts made to convert it into a table syrup have not met with success. Molasses represents the last syrup runnings of the sugar manufacture from which no further crystallization of sugar can be effected, although it still contains about 50 per cent. of sucrose. The uncrystallizability of this large amount of sugar is partly accounted for by the excessive viscosity of the concentrated syrup, but mainly by the mutual solubility of the sugar and non-sugar. The average composition of beet molasses is: sugar 50, ash 10, organic matters 20, water 20 per cent. Owing to the large proportion of ash present, chiefly salts of potash, when used for feeding cattle, it must be given in strictly limited rations, otherwise, by its laxative properties, it is liable to cause scouring, but within these limits it proves to be a highly beneficial fodder, shown by the glossy coat of the animals an indication of health. In order to avoid the inconvenience attending the measuring out and diluting of this viscous material on the farm, molasses is now made up into molasses fodders by absorbing it with peat, bran, chopped straw, etc., which are in a suitable condition for transport and for distribution. Large quantities of molasses are also utilized in distilleries for fermentation to alcohol for the production of silent spirit. The fermentation processes for the production of acetone described and patented by Fernbach and others led to experiments in the United States to develop a process for fermenting molasses to produce acetone which yielded largely ethyl alcohol as a by-product and smaller quantities of propyl and butyl alcohols. By this means the fermentation in

50 to 60 hours gave a yield of 8 to 8·5 per cent. of the sugar as acetone and 20 to 21 per cent. of alcohol.

Another interesting development of the fermentation of molasses is the production of glycerine, an account of which is given by Arthur R. Ling. Inedible Porto Rico "black strap" molasses is fermented by a special yeast at 30° to 32° C., with the addition of sodium carbonate in portions up to a total of 5 per cent. When sufficient carbonate has been added the evolution of gas ceases, a copious precipitate forms, and the yeast lies dormant for a while. The precipitate eventually disappears and fermentation again proceeds. It is essential for the precipitate to form and for the yeast to lie dormant. The alcohol produced covers the cost of the process, so that the glycerine is obtained for nothing. One cwt. of black strap gives 5·5 to 6 lbs. of glycerine. From 25 to 30 per cent. of sugar in the mash is converted into glycerine and 3 per cent. left unfermented. The presence of ammonium chloride favours production of glycerine. Five days are required for fermentation to be completed. In Germany sodium sulphite was used as alkaline substance.

### Recovery of Sugar from Molasses

When for fiscal or other reasons it is not profitable to send molasses to the distillery for the production of spirit, it is treated by one or other of the processes for the extraction of sugar.

**Osmose Process.**—By the process invented by Dubrunfaut, in 1863, the more readily diffusible salts of molasses are separated by osmosis through parchment paper from the less readily diffusible sugar. A series of sheets of parchment paper are arranged as in a filter press, and water and molasses are passed through alternate chambers of the osmose apparatus. The relative rates of flow of water and of molasses are so regulated that the osmose water does not contain more than 1 per cent. of sugar, while the purity of the molasses is increased from about 60 to 70 per cent., so that a crop of sugar may be obtained from it by boiling in the

vacuum pan. The osmose water may be used as a fertiliser, but is often sent to the distillery.

**Elution Process.**—The elution process introduced by Scheibler, in 1865, and subsequently modified by others, consisted in forming tri-calcium sucrate and washing the precipitate with 35 per cent. alcohol. The alcohol in the sucrate and washings was recovered by distillation. The loss of sugar was small and the purity of the saturated sucrate high, but the cost of the outlay and the danger of working the large quantities of alcohol required led to the abandonment of the process.

**Substitution Process.**—In this process, due to Steffen, a very dilute molasses is saturated in the cold with lime to form mono-calcium sucrate, which is soluble in water. The solution is heated to boiling, when tri-calcium sucrate precipitates and two-thirds of the sugar is set free in the liquor. The tri-calcium sucrate is filtered off and washed with boiling water, which does not decompose it. The filtrate is made up to 10 per cent. by the addition of fresh molasses and the process repeated on this substituted mother lye, a fraction of the filtrate being put aside at each operation to prevent the excessive accumulation of non-sugars in it. This part is treated with lime, heated and filtered, and the filtrate utilized as fertilizer.

**Separation Process.**—Steffen's separation process has displaced his substitution process. If a dilute solution of the mono-calcium sucrate is cooled below 15° C. and finely powdered quicklime added gradually with agitation, the whole of the sugar present is separated as a precipitate of tri-calcium sucrate. The precipitate is separated in filter presses and washed with very cold water saturated with lime. The purity of the sucrate is over 96 and is used in the factory for defecation in place of lime.

**Strontia Process.**—The use of strontium compounds for the desugarization of molasses was patented in 1849 by Dubrunfaut and Leplay, but the process was not then adopted. Scheibler worked out the conditions under which the separation of sugar by strontia is possible and patented

wider tube in the centre. In the horizontal form of pan the tubes are arranged in rows vertically over one another with wide spaces between.

**Boiling to Grain.**—It is only juice of high purity that can be readily boiled to grain; juices of lower quality are sometimes enriched by melting with them the unsaleable fine-grained dark sugars from low products. A quantity of juice is drawn into the pan sufficient to cover the lower coil and concentrated to string proof. By the length of the thread formed when a drop of the syrup is drawn out between the finger and thumb it is described as weak or strong proof. For graining thick juice the proof should be rather light. Small quantities of juice are then drawn in suddenly, and owing to the cooling and agitation which ensue crystals are formed in the syrup. Samples are drawn by the proof stick and spread on a glass plate, and from the appearance it is judged whether a sufficient number of crystals have been formed. If so, a larger quantity of juice is drawn in to lower the super-saturation, and this procedure is repeated as often as the proof shows the need for further supplies. As the grain increases in size the panman watches the samples to see that the surrounding syrup is clear and free from small false grain. The indications of the thermometer and vacuum gauge are also noted for guidance, as also the splashes on the sight glasses. The volume of the juice in the pan increases, and as each coil becomes covered, steam is admitted to it, and boiling is continued until grain of the desired size is reached. For raw sugar a coarse, sharp grain is desirable, but for sugar that is to go into consumption direct the grain should be fine. Large grain is obtained by boiling quietly and drawing in large drinks of juice at long intervals during which the crystals grow; finer grain is produced by more vigorous boiling and frequent small supplies of fresh juice. The end of the boiling is indicated by the condition of the massecuite, which should show a large quantity of crystals and only a small quantity of syrup from which the crystals separate distinctly. The massecuite as dropped from the pan should be quite stiff, but crumbly. The time of boiling

a charge is from 6 to 8 hours, and when syrups from a previous operation are drawn into the pan at the end, the boiling may proceed for another hour or two. Boiling does not always proceed normally. Occasionally a quantity of thick froth forms as soon as the juice begins to boil, and there is danger of the whole mass boiling over. This is to some extent overcome by boiling at a lower vacuum and higher temperature, and by the introduction of grease into the pan. The cause is often to be traced to imperfect filtration, or the use of an insufficient amount of lime and faulty defecation. When bad or decayed beetroots have been worked the juice often boils badly and almost ceases as soon as a certain concentration is reached, the contents of the pan remaining motionless like melted fat. The grain forms only with difficulty and grows slowly, and the transmission of heat into the thick mass is so small that caramelization ensues, and the juice becomes dark brown. Slow and difficult boiling has also been attributed to presence of alkali and to calcium salts of arabic acid. The remedy is either to boil merely to string proof or to neutralize with sulphurous or phosphoric acid.

**Control of the Boiling.**—The conduct of the boiling operation is in the hands of an experienced panman who has served an apprenticeship and has learnt from the appearance of the mass how to regulate the super-saturation and the consequent course of formation and growth of the crystals. Boiling to grain was for long considered an art and beyond the control of science. In recent years, however, several forms of apparatus have been introduced to secure this control, among which may be mentioned the brasmoscope of Curin and the apparatus of Claassen. These are slide rule arrangements by which the temperature of the boiling mass in the pan and the vacuum give the percentage of sugar or degrees Brix of the syrup, and this, in conjunction with the degree Brix of a saturated solution of sugar at the same temperature, shows whether the syrup in the pan is or is not saturated or super-saturated. Moreover, it is possible to express the degree of super-saturation as shown by Claassen. If the ratio of sugar to water in a saturated solution be

denoted by S, and the ratio for the syrup in the pan at the same temperature be $S^1$, then $S^1/S$ represents the degree of super-saturation if $S^1$ is greater than S. For graining thick juice the concentration is carried out until this ratio is 1·2 to 1·3, and after the grain is formed, the super-saturation is reduced to 1·05. With lower-class products the ratio requires to be somewhat higher, and in any case, if records are kept of the course of a well-boiled pan, the operation can be safely repeated with the aid of the indicators, preserving the same ratios at the different stages of boiling, and thus avoiding errors of personal judgment. The apparatus, however, only affords reliable indications provided the contents of the pan actually boil.

### Treatment of the Massecuite

When the massecuite is discharged from the pan it is at a temperature of about 80° C., and the syrup is somewhat strongly super-saturated, so that if the crystals were separated at once the syrup would carry off an undue amount of sugar into the lower product. If the mass is allowed to cool quickly the super-saturation increases so much that a crop of very small crystals is formed which pass off with the syrup from the centrifugal machine, being too fine to be retained. To secure this sugar in the first product it is, therefore, necessary to moderate the rate of cooling and to lessen the super-saturation by the periodical addition of somewhat diluted syrup from a previous operation, or even water, as the temperature falls. By this means the viscosity and super-saturation are kept low enough for the sugar to deposit on the crystals already present, and this result is assisted by keeping the whole mass in slow movement until the temperature has fallen to about 50° C. It is not advisable to cool below this temperature, otherwise the mass would become too stiff and not possess the necessary mobility to enable it to rise in the centrifugal machine and form a uniform layer. The whole contents of the pan are let down into a crystallizer which is either a closed horizontal cylinder or a large open trough provided with stirring arms with a

revolution in one or two minutes. The crystallizers are often jacketed so that they may be heated with steam or hot water, or cooled with water during the 12 to 24 hours of crystallizing and cooling. The cooled massecuite may be drawn off in bogies running on an overhead rail, and each carrying 2 to 5 cwt., sufficient for one centrifugal machine, or it is passed along a gutter with a screw conveyor. From the latter each machine draws its charge.

**Centrifugal Treatment of the First Massecuite.**—As soon as the drum of the centrifugal is set in motion, and while it is getting up speed, the massecuite is run in. The whole mass as it falls is gradually thrown against the wall of the basket and rises to form a uniform layer. The sugar crystals are retained, while as the speed increases the liquid syrup passes off through the holes or slits in the metallic cloth of the basket and strikes against the wall of the outer casing, from which it trickles down to the gutter, and is run off as first product drainings. The centrifugal is run as long as syrup comes away, but it is not possible to remove the whole of the syrup in this way, a thin, strongly adherent layer remaining on the crystals. This may amount to some 10 per cent., dependent on the character of the grain. The raw sugar obtained has a net rendement of 88 to 92 per cent. When sugar is required for direct consumption it is necessary to remove the greater part of the remaining adherent syrup which gives to beetroot sugar its unpleasant taste. This is possible when juice of a high degree of purity has been worked and when the defecation has been carefully carried out. After the first drainings syrup has been removed as far as possible in the centrifugal, the sugar is washed with water, steam or with a saturated solution of pure sugar, which are introduced while the drum of the centrifugal is still in motion. The washing removes most of the remaining syrup and yields a white sugar, but it cannot be regarded as a refined sugar which must have undergone solution and purification. The syrup from this operation is, of course, richer than the green, mother syrup first spun off, and is returned to be boiled with the thick juice.

**Crystallization in Motion of the Second Massecuite.**—The green syrup runnings from the raw sugar were formerly boiled to string proof and the concentrated syrup left at rest in shallow iron tanks holding several tons each. After remaining for some days the whole became a mass of fine crystals and syrup, or a deposit of fair-sized crystals with a layer of syrup above. The cold mass was dug out, passed through a pug mill to make it uniform and mobile, and dried in the centrifugal. The syrup drainings were treated in a similar manner to give a third-product sugar and molasses, but required a few months for crystallization to be complete, and the tank rooms had to be kept warm during all this period to render the rate of cooling slow. The third-product sugar was frequently so dark and sticky as to be unsaleable, and had to be re-melted and worked with the raw juice. More recently the centrifugal syrups from the first product are boiled to grain and the massecuite introduced into crystallizers and kept in motion as already mentioned under first-product treatment. Owing to the accumulation of non-sugars in the syrup the grain is more difficult to form in this case and slow in appearing, but it is always possible to grain the syrups unless they are too impure. After grain is formed it is built up gradually into crystals during a boiling lasting 18 to 24 hours, when the pan is then full. It is an advantage if the pan is provided with stirring gear, the rotating arms of which are arranged above and below the calandria. The massecuite is then let down into the crystallizer and kept stirred for 4 or 5 days, while the temperature is allowed to fall from 90° to about 40° C. Syrup or water is added from time to time as may be required to keep the mother syrup from becoming too highly super-saturated, lest false grain should be formed. At the end of this time the mass may be centrifugalized direct. The syrup runnings constitute molasses. Another and earlier method of maintaining the proper degree of super-saturation was to raise the temperature a few degrees as soon as that of the massecuite had fallen about 10°, and to repeat the warming for each similar fall, by passing steam or hot

water through the jacket of the crystallizer. Crystallization in motion not only gives sugars of better quality and higher yield than were obtained by the older method, but the second product is obtained within a few days after the end of the campaign instead of the months required by crystallization in tanks and the expense of keeping the rooms warm all this time.

**Molasses.**—Factory beetroot molasses has an unpleasant saline taste and disagreeable odour, and for this reason is unfit for human consumption, and the attempts made to convert it into a table syrup have not met with success. Molasses represents the last syrup runnings of the sugar manufacture from which no further crystallization of sugar can be effected, although it still contains about 50 per cent. of sucrose. The uncrystallizability of this large amount of sugar is partly accounted for by the excessive viscosity of the concentrated syrup, but mainly by the mutual solubility of the sugar and non-sugar. The average composition of beet molasses is: sugar 50, ash 10, organic matters 20, water 20 per cent. Owing to the large proportion of ash present, chiefly salts of potash, when used for feeding cattle, it must be given in strictly limited rations, otherwise, by its laxative properties, it is liable to cause scouring, but within these limits it proves to be a highly beneficial fodder, shown by the glossy coat of the animals an indication of health. In order to avoid the inconvenience attending the measuring out and diluting of this viscous material on the farm, molasses is now made up into molasses fodders by absorbing it with peat, bran, chopped straw, etc., which are in a suitable condition for transport and for distribution. Large quantities of molasses are also utilized in distilleries for fermentation to alcohol for the production of silent spirit. The fermentation processes for the production of acetone described and patented by Fernbach and others led to experiments in the United States to develop a process for fermenting molasses to produce acetone which yielded largely ethyl alcohol as a by-product and smaller quantities of propyl and butyl alcohols. By this means the fermentation in

50 to 60 hours gave a yield of 8 to 8·5 per cent. of the sugar as acetone and 20 to 21 per cent. of alcohol.

Another interesting development of the fermentation of molasses is the production of glycerine, an account of which is given by Arthur R. Ling. Inedible Porto Rico "black strap" molasses is fermented by a special yeast at 30° to 32° C., with the addition of sodium carbonate in portions up to a total of 5 per cent. When sufficient carbonate has been added the evolution of gas ceases, a copious precipitate forms, and the yeast lies dormant for a while. The precipitate eventually disappears and fermentation again proceeds. It is essential for the precipitate to form and for the yeast to lie dormant. The alcohol produced covers the cost of the process, so that the glycerine is obtained for nothing. One cwt. of black strap gives 5·5 to 6 lbs. of glycerine. From 25 to 30 per cent. of sugar in the mash is converted into glycerine and 3 per cent. left unfermented. The presence of ammonium chloride favours production of glycerine. Five days are required for fermentation to be completed. In Germany sodium sulphite was used as alkaline substance.

### Recovery of Sugar from Molasses

When for fiscal or other reasons it is not profitable to send molasses to the distillery for the production of spirit, it is treated by one or other of the processes for the extraction of sugar.

**Osmose Process.**—By the process invented by Dubrunfaut, in 1863, the more readily diffusible salts of molasses are separated by osmosis through parchment paper from the less readily diffusible sugar. A series of sheets of parchment paper are arranged as in a filter press, and water and molasses are passed through alternate chambers of the osmose apparatus. The relative rates of flow of water and of molasses are so regulated that the osmose water does not contain more than 1 per cent. of sugar, while the purity of the molasses is increased from about 60 to 70 per cent., so that a crop of sugar may be obtained from it by boiling in the

vacuum pan. The osmose water may be used as a fertiliser, but is often sent to the distillery.

**Elution Process.**—The elution process introduced by Scheibler, in 1865, and subsequently modified by others, consisted in forming tri-calcium sucrate and washing the precipitate with 35 per cent. alcohol. The alcohol in the sucrate and washings was recovered by distillation. The loss of sugar was small and the purity of the saturated sucrate high, but the cost of the outlay and the danger of working the large quantities of alcohol required led to the abandonment of the process.

**Substitution Process.**—In this process, due to Steffen, a very dilute molasses is saturated in the cold with lime to form mono-calcium sucrate, which is soluble in water. The solution is heated to boiling, when tri-calcium sucrate precipitates and two-thirds of the sugar is set free in the liquor. The tri-calcium sucrate is filtered off and washed with boiling water, which does not decompose it. The filtrate is made up to 10 per cent. by the addition of fresh molasses and the process repeated on this substituted mother lye, a fraction of the filtrate being put aside at each operation to prevent the excessive accumulation of non-sugars in it. This part is treated with lime, heated and filtered, and the filtrate utilized as fertilizer.

**Separation Process.**—Steffen's separation process has displaced his substitution process. If a dilute solution of the mono-calcium sucrate is cooled below 15° C. and finely powdered quicklime added gradually with agitation, the whole of the sugar present is separated as a precipitate of tri-calcium sucrate. The precipitate is separated in filter presses and washed with very cold water saturated with lime. The purity of the sucrate is over 96 and is used in the factory for defecation in place of lime.

**Strontia Process.**—The use of strontium compounds for the desugarization of molasses was patented in 1849 by Dubrunfaut and Leplay, but the process was not then adopted. Scheibler worked out the conditions under which the separation of sugar by strontia is possible and patented

the processes. In the older method crystals of strontium hydroxide, $Sr(OH)_2$, $8H_2O$, are added to a hot dilute solution of molasses until the solution is saturated, and it is then heated to boiling. The precipitate of di-strontium sucrate is only stable whilst hot; on cooling, it decomposes with separation of sugar. It must, therefore, be filtered hot and washed with boiling water. The washed sucrate is stirred to a cream in cold water and crystals of the hydroxide separate, leaving a nearly pure solution of sugar containing only a small quantity of strontium hydroxide. This is precipitated as carbonate by passing in a stream of carbon dioxide. In the newer process the sugar is precipitated as the mono-strontium sucrate. The molasses is run into a hot (75° C.) solution of strontium hydroxide. If the solution of mono-strontium sucrate thus formed were left to cool at rest, strontium hydroxide would crystallize out; but if it is kept stirred and crystals of the mono-sucrate added, the whole of the sugar separates out as a crystalline meal of mono-strontium sucrate. This is separated from the lye by suction filters, filter presses, or in centrifugals, and washed with cold water. To obtain the pure sugar solution from it, the mono-sucrate is dissolved in hot water; on cooling, most of the strontium hydroxide crystallizes out, the solution can then be treated with carbon dioxide, filtered and worked up into sugar.

The strontia process is perhaps the most complete, but also the most expensive of the desugarizing processes. About 200,000 tons of sugar were recovered by this process in Germany in 1912–1913.

The Preliminary Report of the Empire Sugar Supply (Technical) Committee was presented before a Conference at the annual meeting of the Society of Chemical Industry on July 16, 1919. The Report gives numerous statistical tables of the production and consumption of sugar throughout the world with particular reference to the supply and needs of the British Empire. The sugar produced within the Empire is chiefly derived from the cane, and while the consumption amounts to 6,250,000 tons, the total production

within it falls short of this by about 2,500,000 tons. It was a matter of debate how this deficiency could be supplied so as to make the production of sugar within the Empire self-supporting. Many speakers favoured India as the country which could fulfil this want most readily and suitably. The subject of the establishment of a beet sugar industry in the United Kingdom was also considered. Beets of sugar content and purity equal to those produced on the continent can be grown here, but there is still much to be learnt about the cultivation, and co-operation between the farmers and manufacturers will be necessary to ensure the supply of roots needed for success in working a factory. There is little fear that growing beets will lessen the acreage under wheat and other cereals, and the residues will form useful food for cattle. The world's production of beetroot sugar in 1913 was 8,758,900 tons, or 46·96 per cent. of the total production of cane and beet together. The cane production at the same time was 9,894,200 tons.

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## Section III.—Sugar Refining

Sugar refining is restricted to the preparation from raw sugar of a clean, generally white sugar fit for consumption in forms required to suit the convenience and taste of the consumers. The more usual forms are loaf-sugar, cubes, crystals, granulated, and moist sugars of fine grain as white and yellow pieces.

The residual molasses is either made into treacle or other table syrup as golden syrup, or sold to the distillery or used for making molasses fodder.

**Raw Sugars.**—Raw beet sugars are unsuitable for direct consumption owing to their nauseous taste, due to the presence of salts and organic non-sugars. Raw cane sugars, although often very impure, have a pleasant aromatic taste. They frequently contain numbers of living and dead acari (the sugar-itch "insect") as well as crowds of bacteria, moulds and yeasts and vegetable debris, and need to be refined to obtain a wholesome product.

The whole of the sugar present in raw sugars is not recoverable as such in the ordinary process of refining, the salts and organic matters rendering a portion of the sugar uncrystallizable, so that this remains in a final syrup product, refinery molasses.

The raw sugars are valued on the net rendement, five times the ash (salts) being subtracted from the polarization on the assumption that so much sugar is rendered uncrystallizable thereby. A deduction is also made for reducing sugars present. The price of raw beet sugars is fixed on a basis of 88 per cent. net, with a valuation for

variations above and below this. Second quality beet sugars are classed as 75 per cent. net.

**Affination.**—Raw sugars were formerly melted direct, and preference was given to sugars of good grain and of a clear, pale colour. The liquor then contained all the non-sugar of the raw. It is now usual to wash the raw sugars whereby the greater part of the molasses adhering to the crystals is removed and the non-sugars contained in it also pass off into this affination syrup. The sugar is mixed to a mash with hot syrup from a previous operation, and the mass is spun in the centrifugal machine. The washed sugar is melted, while the syrup is boiled to obtain a crop of sugar and molasses. Raw beet sugars of good sharp grain are sometimes subjected to a systematic washing in tanks by a series of syrups of graduated purity so as to obtain finally a more or less white sugar. This process, due to Steffen, although known previously, is used abroad. Soft-grained, smeary sugars unsuitable for washing in this way are, according to Langen, heated in a vacuum pan to melt the fine grain and the mass then subjected to crystallization in motion in a Bock apparatus, after which the sugar is in a condition for the Steffen washing process.

**Melting.**—The washed sugar from the centrifugals is dissolved or melted in a pan (blow-up) provided with stirring gear and heated by steam to 90° C. until the liquor attains a density of 55°–60° Brix.

When raw unwashed sugars were melted defecants were often employed at this stage to aid in the separation of the colouring matters and other impurities, and especially to facilitate filtration, and with washed sugars a little milk of lime is frequently added to secure a faint alkalinity of the liquor so that there may be no danger of inversion of sugar during subsequent operations. Cane sugars are much more difficult to filter than beet, and the addition of porous and granular substances such as diatomite greatly assists filtration.

**Filtration.**—When the liquor leaves the blow-up it runs over a strainer of perforated metal or wire to remove the

coarser suspended impurities, and is then either pumped direct to filter presses or to a tank from which it flows at a few feet head of pressure through one of the various forms of pocket or bag filters, or in some cases through a sand filter. It is essential that a clear, bright liquor is obtained for passing to the char cisterns to be decolorized.

**Filtration through Animal Charcoal.**—The char cisterns are cast- or wrought-iron cylinders 5 to 10 feet in diameter and 15 to 20 feet in height. The bottom is slightly conical and has a perforated wooden false bottom covered with a sheet called the blanket to prevent the grains of char passing through with the liquor. The cisterns each hold 10 to 40 tons of char. The liquor which has passed through the column of char of suitable grist issues practically colourless at first, but gradually becomes more coloured after running some time. The char absorbs a certain small amount of inorganic and organic non-sugars, but the decolorization is of most importance. Decolorization by char is evidently a surface action, and not chemical, for the absorbed colour can be removed from the char by treatment with alkali. The decolorization is greater when the solutions are kept slightly acid, but there is always the danger of inversion of sugar if the liquor is allowed to become acid. The more solid and dense bones give the best char, which has a closeness and lasting quality, and does not easily wear to dust by the continual handling to which it is subjected. The cartilage of bones is rich in nitrogen, and permeating the whole structure there is nitrogen throughout the char, and the high decolorizing power is often attributed to the nitrogen present, so that attempts to manufacture artificial chars have been frequently made by soaking porous materials with gelatine or glue before carbonizing. Good char is of a deep, dull black colour and is highly porous. When dry it contains 78 per cent. of calcium phosphate, 10 of calcium carbonate, 8 to 10 of carbon, and small quantities of soluble salts as well as calcium sulphate and sulphide, magnesium phosphate and silica. It is hygroscopic, and is usually sold on a basis of 8 per cent. of moisture. When the char no

longer decolorizes sufficiently it is washed with hot water, drained, and removed to be passed through a series of pipes heated to incipient redness in a kiln. It is then said to be revivified. Char may be revivified a great many times before its decolorizing power deteriorates so far that it is considered to be useless. During the reburning absorbed organic matters are carbonized, and the carbon thus produced, which has little or no decolorizing power, in course of time blocks up the pores. Weinrich has introduced a method of burning off this added, useless carbon by passing the char through an open rotating cylinder heated to a temperature sufficient to burn off the added carbon without attacking the original carbon of the char. The decoloring power of the char is considerably improved by this treatment. Bussy found that when char or char carbon was ignited with potassium carbonate and the salt washed away, the decolorizing power was increased about ten times. Recently a number of vegetable decolorizing carbons have been produced by heating sawdust or other cellulose material with lime, or with calcium, magnesium, or zinc chloride, and dissolving away the lime or salts. These have a much greater decolorizing power than char or char carbon, but owing to their high price, and the difficulty of manipulation as compared with the way in which char is worked, have not yet become established in practice.

**Boiling to Grain.**—The decolorized fine liquor collected from the char cisterns is boiled in the vacuum pan, much as has been described under the boiling of raw sugars, only that the greatest care and attention is directed to secure a grain of uniform size without the presence of small grain or masses of coherent grains called rolled grain. When a larger-sized crystal is desired, part of the contents of a pan is dropped and boiling resumed until the crystals present have attained the desired size. When the whole contents of the pan is discharged into a heater it is kept stirred while portions are drawn off to separate the sugar from the mother syrup in the centrifugal machine.

**Drying the Massecuite.**—The magma of crystals and

syrup is spun in the centrifugal machine, and as soon as the bulk of the syrup has run off, water is sprayed on the sugar in the machine, a small quantity of ultramarine or other suitable blue being added to the water to correct any slight tinge of yellow in the sugar. The syrup formed by this water by dissolving a little of the sugar is often collected separately. The sugar is removed while still warm, and has a moisture content of 1 per cent. or less. It is then passed over sieves to remove any unavoidable lumps which are sometimes formed through sugar adhering to the steam coils.

**Granulated Sugar.**—For granulated sugar the sieved sugar is passed through a slightly inclined revolving cylinder heated by an inner cylinder through which steam passes, and provided with blades at its inner periphery which lift the sugar and allow it to fall over the heated drum so that it leaves the granulator with a moisture content of 0·05 per cent. or less, after which, when cooled, it is weighed into bags.

**Cube Sugar.**—The old-fashioned loaf sugar has now been almost entirely displaced by the production of cube sugar. The massecuite is filled into moulds consisting of a series of metal plates separated by a distance equal to a side of the cube to be produced. As soon as the mass has set a number of these moulds are arranged inside the centrifugal machine and held in place by blocks of triangular section, and the whole is then spun together. As soon as the mother syrup has run off, a quantity of a pure syrup—cleare—is poured in to displace the last portion of mother syrup. The moulds are then removed and the plates or slabs of sugar are placed in a hot room to dry and harden, the syrup left between the crystals solidifying and cementing the crystalline grains firmly together. The dried slabs are then placed on a cutting-machine, when a series of cuts are made which crack or cut the plates into the form of cubes which roll over a screen to allow any small broken pieces to fall through.

The syrups from the boiling of granulated cubes and crystals are boiled again to give other crops of sugar, until finally the syrup remains which constitutes refinery molasses

and may be made either into a low quality table syrup or treacle.

**Preparation of Invert Sugar and Saccharum.**—Any of the intermediate syrups from the sugar may be taken according to quality for the preparation of invert sugar. For this purpose the diluted syrup is heated in a wooden or lead-lined tank with a quantity of dilute sulphuric acid until samples withdrawn show by the polariscope test that the sugar is completely inverted. The syrup is then run out into the neutralizing tank and made neutral by the addition of powdered chalk. The syrup is then pumped through a filter press to remove the sulphate of lime, and the clear liquor passed through char to improve the colour, after which it is concentrated in the vacuum pan to the required density. If the resulting syrup when cooled is stirred it forms a honey-like paste, and when filled into pails will gradually set to a firm mass of saccharum. The operation is aided by addition of a portion of solid saccharum from a previous operation.

**Table Syrups.**—An intermediate syrup is added to a portion of invert syrup, or, in the case of low cane sugars, even without any addition of invert is carefully filtered and decolorized by passing through char and then concentrated to the required density in the vacuum pan. It is necessary that the proportions of cane sugar and invert be properly adjusted that the syrup may remain bright and clear, and not grain out.

**Refinery Molasses.**—The lowest syrups from the boiling of washed sugar is more correctly a comestible treacle. The affination syrup, after yielding one or more crops of sugar, forms a true molasses, and is sent either to the distillery or to be made into cattle food.

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*Journal of the Society of Chemical Industry.*

## Section IV.—MINOR SOURCES OF SUGAR

**Sorghum Sugar and Syrup.**—The value of Sorghum cane, *Andropogon*, as a source of sugar has long been recognized in Africa and China, and attention having been directed to this plant, the United States Department of Agriculture cultivated about thirty varieties of sorghum with a view to the profitable extraction of sugar therefrom. Of these the Early Amber was the variety mostly in favour with planters in Minnesota and the north-west. It derives its name from its early ripening and the bright amber colour of the syrup made from it. It is very rich in saccharine matter, the juice containing 14·6 per cent. of sucrose. Other varieties are the Honduras, White Liberian and Chinese or Sumach cane. Although State aid in the shape of bounty was granted to favour the industry the use of sorghum as an economical source for the production of cane sugar had to be given up because the invert sugar, starch, and gums present in the juice prevented crystallization of the sucrose. On the other hand, the concentrated juice forms a useful syrup, and the seed of the cane a food for pigs and cattle of feeding value equal to Indian corn. The juice expressed from the cane by mills, horse-power, or steam, is defecated with a little lime, and after clarifying is evaporated to the required density. One hundred gallons of juice should be obtained from 1 ton of stripped cane. This, when concentrated to 38° to 42° Baumé, produced a syrup of good keeping quality, wholesome, highly nutritious, and palatable to those who have become accustomed to its peculiar sorghum flavour. It forms a staple dietary food in the northern and central portion of the States. The estimated production in the United States was 17 million gallons in 1900, with a yearly

increase of about 15,000 gallons at 19 cents per gallon. An analysis of a syrup showed 36·7 per cent. sucrose, 26·6 per cent. reducing sugars, 4 per cent. ash, and total solids, 76 per cent.

In a recent report to the Academy of Agriculture, Paris, André Piédalia, a French chemist, drew attention to the marvellous utility of the sorghum grass, which is capable of supplying sugar, fodder, paper, flour, and dyes for textiles. The plant is indigenous to equatorial Africa, and was carried thence to Egypt, India, and China. In the fifteenth century it was growing in Italy, in Genoese and Venetian territory. In 1850 it was recommended to agriculturists in France, but did not arouse sufficient interest. In suitable climates it gives a good yield of sugar, in China 0·7 ton per acre. The fibres yield 2 tons of paper pulp per acre, and furnish a paper of dazzling whiteness. The leaves serve admirably for fodder and the roots for the production of alcohol; the seeds supply starch, nitrates, and fatty matter constituting a flour of good taste easily mixed with bread stuffs, and are enveloped in gluten from which colouring matters may be prepared.

**Maple Sugar and Syrup.**—Maple sugar is manufactured from the sap of the *Acer saccharinum* or sugar maple. When the sap begins to flow at the end of winter, before the buds of new leaves have developed far, the trees are tapped 3 or 4 feet from the ground with a half-inch auger, the hole made is about an inch deep, and a perforated plug is inserted to collect the sap in a light tin can. Two or three holes or notches are made in each tree, and fresh places made the following year. The trees are not tapped until 25 years old, but can then go on yielding sap for several years without harm. The average yield is 10 to 25 gallons each season of 4 to 6 weeks; 1 lb. of sugar is obtained from 4·5 to 5 gallons of sap. The sap is extremely bright and clear and needs little or no clarifying. It is generally evaporated in iron boilers 6 by 2·5 feet, and 6 inches deep, but copper pans are better. It is usually strained during evaporation, and a little lime added to correct acidity, and milk or white of egg

added to clear it. After straining and skimming the concentrated syrup is poured into moulds to crystallize on cooling. The skimmings formed during concentration are due to coagulation of the albuminoids present in the sap. Often a deposit of calcium malate and silica is formed on the bottom of the pan. Genuine maple sugar products are distinguished from cane products by the large amount of precipitate formed on adding lead acetate, due chiefly to the malic acid present. Both the sugar and syrup have a most attractive characteristic flavour and honey-like odour. It is stated that a docoction of hickory bark will communicate this flavour to cane syrup and sugar, but there are strict regulations against admixture with cane sugar. The mass of concrete sugar from the moulds is never refined, as this would remove the flavour and odour which give the chief value to this product. The syrup is evaporated to a water content of 25 to 30 per cent., and usually contains 63 to 64 of sucrose, 1·5 of reducing sugars, 0·5 of ash, and 4 per cent. of organic matters. The output of maple syrup in the United States in 1900 was about 2 million gallons. Little is made except in the New England States of Vermont, New York, Ohio, and Indiana. In Canada it is made chiefly in Quebec, Ontario, New Brunswick, and Nova Scotia; the amount in 1901 was 17,804,825 lbs. of a value of 1,780,482 dollars. Canada produces three-sevenths of the world's consumption of maple sugar, but is capable of producing 5 or 6 times this quantity. The Canada Food Board, in conference with the leaders in the maple sugar industry, recently made efforts to increase the production of both syrup and sugar. The total exports of maple sugar increased from 2,800,000 lbs. in 1917 to 3,550,000 lbs. in 1918.

**Palm Sugar.**—Many kinds of palm yield a saccharine juice which is utilized in the East for the production of crude sugar. The date palm (*Phœnix Sylvestris*), the palmyra (*Borassus flabelliformis*), the coconut (*Cocos nucifera*), the gomuti palm (*Arenga saccharifera*), the nipa (*Nipa fruticans*) and the kittool (*Caryota urens*). Out of 3,000,000 tons of crude sugar of India's annual output it is estimated

that 10 per cent. is derived from palms and about 4 per cent. from the date palm cultivated for the purpose in Bengal. Palm sugar is made by an incision in the soft upper part of the stem and collecting the juice in earthenware pots. It is then concentrated so that it sets on cooling to form *gur* or *jaggery*, a dark-brown sugar in considerable demand in India. Gur is sometimes treated in native refineries by covering it with a layer 4 or 5 inches thick of the water weed *Vallisneria spiralis*. The molasses drains through the basket to a pot below. Fuel for concentrating the juice is a difficulty. The pots are smoked inside to prevent fermentation, but lime-washing would be better. Although the juice is water white as it exudes, it darkens rapidly on boiling; to lessen this, lime juice is added. The natives obtain 31 per cent., but centrifugal machines would yield 59 per cent. The chance of establishing small central factories is nullified by the cultivators refusing to sell at a reasonable price. 2·3 tons of gur can be obtained from an acre of palms, and this can be increased.

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## SECTION V.—CARAMEL

WHEN sucrose is heated above its point of fusion it becomes coloured and ends by becoming transformed into a brown mass, caramel. Glucose also yields a like product. Caramel is evidently a carbohydrate of high molecular weight and a condensation product of sucrose or glucose. From cryoscopic determinations and analysis of its barium compound its formula has been given as $C_{125}H_{188}O_{80}$.

**Properties.**—Caramel is found in commerce as a concentrated solution or in solid masses or in powder as coffee essence. The solid forms an amorphous, reddish brown, brittle mass which is porous and highly deliquescent. It is completely soluble in water and partly soluble in alcohol. It has a bitter taste due to the presence of assamar, and is not fermented by yeast. Its melting-point is about 135° C. It reduces the salts of the heavy metals and is precipitated from aqueous solutions by baryta and by neutral lead acetate, as well as by paraldehyde in alcoholic solution.

**Manufacture.**—Caramel was originally made by heating cane sugar, and that used for colouring spirits is still generally produced in this way. The sugar is heated in an open metal pan over a gas fire with constant stirring, till the whole mass melts, turns brown, and then suddenly froths up, when the heating must be stopped at once. If heated too far it is inclined to char. Water is cautiously added, and when diluted to the right point the caramal is filtered through wire gauze to remove the carbon and insoluble products. Glucose has now in great measure replaced cane sugar in the manufacture, but unless the glucose is comparatively free from dextrin the product is not so readily soluble in spirit. Potassium or sodium hydroxide or carbonate is heated with

glucose over a fire until irritating vapours commence to appear, the heat is then moderated and the mass stirred continuously until samples show the required tint, when water is immediately added to stop the further change. Ammonia or ammonium carbonate is also used.

**Uses.**—The use of caramel for colouring beer is of comparatively recent application, due to the replacement of part of the malt by sugar or raw grain or less highly kilned malts. The colour is more easily adjusted with caramel to a constant standard. Caramel is also used to colour spirits, liqueurs, vinegar, gravies, and as a coffee surrogate when mixed with suitable aromatics and fillings.

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## PART III.—ALCOHOLIC FERMENTATION—BEER

FRUIT juices on keeping readily undergo a change during which the liquid seethes or ferments and acquires a palatable flavour and an exhilarating physiological property with loss of sweetness, and this process doubtless led to the first use of fermented beverages. Saccharine juices from other parts of plants or honey would in course of time also be utilized in a similar manner. Germinating seeds of cereals become sweet from the conversion of starch into sugar, and the extract therefrom would also furnish material for a like use; in fact, there is evidence that malted grain was used in prehistoric times even in the Stone Age. Where saccharine juices from plants were not available the milk of animals was fermented to produce Koumiss by the Tartars and Kephir in the Caucasus. In the warm parts of southern Europe the juice expressed from grapes is fermented to produce wine, in the colder northern parts apples are crushed and the fermented juice gives cider, but the bulk of fermented beverage in these parts is made from malted barley to produce beer.

**Barley.**—Although the seeds of various cereals have been used at times for malting, centuries of experience have shown that barley is the most suitable grain for the production of good beer, owing to the quantity of starch it contains, the activity of the diastase formed when malted, and the strong husk which allow the worts to be readily drained. Malts from wheat and oats are still made to some extent, have special qualities, and are generally used only with a large proportion of barley malt.

When each of the flower-bearing spikelets which grow alternately on each side of the ear are fertile six-rowed barley is the result, but most of the malting barley grown in this country is the narrow-eared two-rowed barley, ***Hordeum distichum***, the two principal varieties of which are known as the Chevallier and the Goldthorpe. Long experience is required to decide from samples which barley is most suitable for malting purposes. The grains chosen should be of large and uniform size, plump, of pale colour and of sweet smell without the objectionable odour due to mouldiness. The number of cracked and broken grains should be few, and the barley should not feel cold and damp to the hand, but should flow easily through the fingers. When a grain is bitten through it should appear white and floury, not glassy or steely. The vitality of the grains is improved on storing for a time due to gradual drying, and the skin is then somewhat crinkled. The vegetative energy is tested by finding the percentage of grains which germinate when kept for three days or longer in a moist chamber.

**Malting.**—The operation of malting consists in allowing the grains of barley, which have been reduced to uniform size by screening or sifting, to undergo a restricted germination whereby the insoluble starch of the grain is converted into soluble fermentable sugar and then checking further germination by the application of heat. The sifted barley is run gradually and stirred into the requisite quantity of water not below 50° F. and steeped for fifty hours or longer until a sufficient amount of water has been taken up, and the whole contents of the grain are wetted without being sodden. The water is drained to waste and the steeping may be repeated. The action is not merely mechanical, for dust, bacteria and the spores of moulds are washed away and a certain amount of organic matter and soluble salts are also removed. The steeped and drained grains are then run out into a couch or frame and left for a day. One side of the frame is then removed and the grains spread over the floor so as to cover a greater or less area according to the weather to keep the temperature not over 60° F. and avoid the accumulation of

carbonic acid given off by respiration. The "floor" is turned twice daily with broad flat shovels, and the operation repeated during five to eight days or more. The grain soon begins to sprout or "chit," forming a white protrusion of several curly white bushy rootlets, and the plumule, or "acrospire," is visible as a swelling beneath the skin. During this period it is frequently necessary to sprinkle the grain with water to maintain the requisite degree of moisture. As soon as the plumule has grown about three-quarters of the length of the grain further growth is stopped by drying the grain, or withering when it is ready to be dried on the kiln. At this stage the corns should be crumbly and not pasty and the rootlets dry and shrivelled. In pneumatic malting the grains are not spread on the floor, but rotated in drums supplied with moistened air. Considerable floor space is saved by this method.

The malt is dried on the kiln for three or four days, a moderate heat only being applied until the malt is dry. By drying on the kiln the malt acquires a pleasant empyreumatic flavour. The floor of the kiln is of tiles or woven wire, and the source of heat is the combustion gases from anthracite coal or coke, which should be low in sulphur and arsenic. Pale malt, intended for pale and stock ales, is cured at a lower temperature than high-dried malt, which is suited for the production of beers of a sweeter character and fuller nature, the so-called quick-running ales, for which the temperature is raised to 230° F. On the continent malt is kilned by hot air alone, the products of combustion not being allowed to come into contact with the material.

**Diastase.**—The larger portion of a grain of barley is formed by the endosperm in which the starch which constitutes the reserve food material for the young plant is stored up; the smaller part at the end of the grain is the embryo, and separating these is a cellular layer, the scutellum, from which under the influence of the living embryo during germination, an enzyme, diastase, is secreted which converts the starch of the endosperm into soluble sugar capable of being absorbed and utilized in the growth. The starch,

however, is enclosed as granules in the cells of the endosperm, and the diastase as a proteid cannot pass through the walls of the cells; another enzyme, cytase, is secreted which dissolves or changes the cell walls so that they become permeable, and the starch is thus exposed to the action of the diastase. The endosperm is surrounded by a layer of cells, the aleurone layer, containing proteins, which also contributes to this action. Moreover, there are other enzymes which convert the protein contents of the cells into diffusible and soluble substances to help in the nutrition of the growing plant and supply material at a later stage for the nutrition of yeast during fermentation. These various zymases are catalysts of an albumenoid nature, and minimal quantities serve for converting comparatively large quantities of the special substances on which they act. They are only secreted by the living plant; dead corns produce no diastase. They are all sensitive to the destructive action of heat, but not equally so; but are able to withstand higher temperatures when dry. Diastase is contained in air-dried malt, less in kiln-dried, but even this contains more than sufficient to convert all the starch of the grain. Cytase is also present in air-dried green malt, but not in kiln-dried, since it is destroyed at 60° C., at which temperature the activity of diastase remains.

**Supplementary Materials.**—Other materials containing starch used along with malt are maize, oats and rice. The maize and rice are usually torrefied, or more or less gelatinized and crushed between rollers to form flakes, and in this material there is over 75 per cent. of starch with only small quantities of soluble nitrogenous products. Cane sugar, invert sugar and glucose also supply fermentable material without unduly increasing the nitrogenous constituents. Highly coloured malts are made by kilning the malted barley at a high temperature or roasting barley or malt as coffee is roasted. These brown or black malts are used for stout and porter. Caramel is also often used.

**Preparation of Grist.**—The malt from the kiln is ground or, rather, crushed by passing between a pair of steel rollers

so as to form the grist. The disintegration of the husk is avoided as far as possible so that the soluble matters may be readily drained off after mashing, while the interior of the grain is set free so that the starch may be readily acted upon by the diastase when water is added in the mash tun.

The methods of grinding the malt between stones, or of cutting and tearing it with steel cutting mills in the way that coffee is ground, do not yield so satisfactory a product. Malt, when finely ground, is liable to set during mashing and form a mucilaginous magma from which much of the liquor cannot be removed except by prolonged washing, thus rendering the wort dilute, and therefore more liable to suffer from acetification. When the grain is torn or sliced, water will not penetrate thoroughly during the time usual in mashing, and some of the material is unacted on and wasted. It is only by the use of rollers that the malt can be properly prepared, so that the crushed material allows of the mash being easily racked off, leaving but little extract in the grains. The grist mill usually consists of two smooth steel or, better, iron rollers, one of double the diameter of the other, the larger one driven, and the smaller roll revolving by the friction of the malt passing between the two. The distance apart of the rolls is adjusted by a screw to suit the size of the malt being crushed. The grist formed is raised by an elevator consisting of an endless belt provided with buckets, termed a " Jacob's ladder," and delivered to the grist hopper ready for mashing.

## SECTION II.—MASHING

THE crushed malt or grist and "liquor," as water is termed in the brewery, are mixed either in the mash tun or in a preliminary mash mixer. The water is previously heated so as to get the necessary temperature limits for the changes which occur. The mash tun is a cylindrical covered vessel, provided with a perforated false bottom and with revolving rakes, and a series of pipes for drawing off the clear wort. There is also a sparger, consisting of a pipe perforated throughout half its length on one side, and along the other half on its other side; this pipe is made to revolve so that when water is supplied to it the whole surface of the "goods" are thoroughly wetted. Liquor at a high temperature is run in and allowed to remain until the vessel is sufficiently warmed, when the water is run off. The grist and water are then run in, the proportions being so regulated that the mash will possess the proper consistency, the rakes meanwhile being kept in motion until the whole is thoroughly mixed. The contents are then left at rest for about two hours until tests show that all the starch has been converted, when the clear, bright wort is drawn off into the under-back or copper. While the wort is running off sparging is continued so as to avoid any tendency to set and obstruct free drainage. This will form a second mash. After this is run off the material should be sufficiently exhausted, and is removed as brewers' grains to be sold to farmers for fattening cattle.

**Nature of the Changes during Mashing.**—During the mashing process the starch of the grist is acted upon by the diastase and the starch molecule broken down (page 6) into maltose, dextrin and intermediate malto-dextrins, one of the

latter being practically unfermentable and hence termed stable dextrin. The relative proportions of the malto-dextrins which contain relatively few amylin groups and a large number of maltose groups, and therefore more readily fermentable, and those in which the reverse is the case is determined by the temperature at which the malt has been kilned and mashed, and on this will also depend the class of beer produced therefrom, the flavour and character of the beer being due as much to these extractives as to the alcohol present. A pale ale, free from excessive sweetness, must contain more of the less readily fermentable bodies in order to provide for cask fermentation and the constant slow production of carbonic acid to keep it in condition, while a mild, quick-running ale must be sweet and, therefore, made from wort rich in free maltose and malto-dextrins approaching thereto, and readily reach condition in the cask. These required conditions are obtained within the narrow range of 145° to 155° F., at which mashing is carried out, the diastase being extremely sensitive to slight variation of temperature; if mashing is done near the lower temperature the wort is suitable for mild and sweet ale, at the higher, for stock beer. When raw grain is used in conjunction with malt it requires to be gelatinized so that the starch may be acted on by diastase during mashing, or it may be treated in a converter with a little pale malt at a high temperature to form soluble starch, otherwise it would form a viscous mash. The above infusion method of mashing is best suited for brewing English beers, but as all malt is liable to contain some unmalted grain this portion remains unacted on during the process and is lost as regards the production of beer. On the Continent and in the United States the decoction method of mashing is used. The malt is first mixed with water at a lower temperature than in the infusion method and the mash then heated in successive stages nearly to the boiling-point; or, a portion of wort is run off and the contents of the mash tun heated nearly to boiling-point by steam, then cooled to mashing temperature, and the portion of wort containing active diastase returned to it and mashing allowed to

complete. By this means any unmodified corns in the malt are gelatinized and rendered capable of being saccharified so that the extraction is more complete. The density of the wort is taken by a special form of hydrometer giving the original gravity of the beer to be brewed from it.

**Influence of the Water used for Brewing.**—Water is one of the most important requirements for brewing, and a constant and abundant supply of suitable clear water is therefore an indispensable condition for a brewery. The waters best adapted for brewing purposes are those which are of organic purity and not contaminated with poisonous metals, or more than a trace of iron, and contain inorganic salts within certain limits. Experience has proved that some waters are more suitable for the production of ales of a particular character, but not for those of another class. The Burton pale ales and the black beers of Dublin and London furnish cases in point. The Burton water contains large quantities of calcium sulphate (over 70 grains per gallon), while the Dublin water contains only a small quantity of calcium sulphate, but a somewhat larger amount of calcium and magnesium bicarbonates, which are almost completely precipitated on boiling, leaving the water soft. Water most suitable for brewing the best mild ales contains intermediate quantities of calcium sulphate (5 to 7 grains per gallon) but a large amount of sodium chloride (35 grains per gallon). On the addition of the necessary amounts of the salts lacking it is possible in many cases to make certain waters more suitable than they would otherwise have been for a specific purpose. The addition of calcium sulphate, magnesium sulphate and calcium chloride in proper amounts to water containing a deficiency of these salts will make it suitable for use in brewing good pale ale.

## Section III.—BOILING AND HOPPING

The sweet wort from the mash tun is collected in the under-back, a copper vessel provided with a steam coil to heat the wort immediately it enters to such a temperature as to render the diastase inactive and prevent further conversion of the malto-dextrins. From the under-back, or sometimes directly from the mash tun, the wort is run by gravity or pumped to the boiling-copper, which may be arranged to take the whole of the wort brewed, or the stronger and weaker worts are boiled separately. The copper may be heated by fire or steam, and boiling is continued for about two hours, during which the hops are added in portions, the best hops being added last, just before the bitter wort is run off. As a result of the boiling the wort becomes completely sterilized, the various bacteria, moulds and yeasts on the barley and malt being killed, some of the proteid matters are coagulated, the action of the diastase is arrested unless this was done in the under-back, the aroma and flavouring constituents of the hops are extracted, as well as the antiseptic resins, and tannin, and the wort becomes more concentrated.

**The Chemical Constituents of the Hops.**—The portion of the hop plant used by brewers is the strobile or cone, the aromatic smelling blossoms of the hop bine. The strobiles of the female plant only are used; these consist of partly overlapping scales or petals which are of two kinds, one seedless, the other containing the fruit or seed. At the bases of the scales is a yellowish powder, lupulin, spoken of by the brewer as "condition," since it contains the flavouring essential oil, the resins, the bitter principles and other ingredients to which hops owe their value. The essential oil was shown by Chapman to consist of two hydrocarbons,

*myrcene*, which possesses a penetrating odour and readily changes into a resin, and *humulene*, which has very little odour and is not so prone to change, as well as other oxygenated bodies in small amount, but which contribute largely to the odour of the oil. The oil is only very slightly soluble in water, but somewhat more so in the dilute alcohol of beer and is yet sufficient to confer its flavour on this. The oil is volatile with steam, and on boiling the wort some of the aroma is lost. It is for this reason, in order to minimize the loss, that the hops are added in portions to the wort, the best hops last just before the wort is run off. The resins and bitter substances not only confer flavour but act as antiseptics and prevent the ready development of bacteria in the beer. There are several resins present, but the chief distinction made is into the soft and the hard, the former of which is preservative, and being unstable readily passes into the less effective hard form. It has been found that if hops are stored at a low temperature the rate of deterioration is much lessened. The bitter principles of the hop are due to certain acids closely allied to the resins. A certain amount of tannin is extracted from hops during the boiling, and this contributes to a small extent in precipitating proteins and thus enabling them to be removed.

**Cooling and Refrigerating.**—The boiled wort is run into the hop back, a wooden vessel provided with a perforated false bottom, to keep back the hops which act as a filter and allow the clear wort to flow through. The hops are sometimes sparged to remove the wort retained, or they are passed through a form of press to squeeze out the wort. The filtered wort is pumped up to the cooler, a large shallow rectangular vessel in which the wort was formerly cooled to the temperature suitable for fermentation. Although the tank is usually placed in a room at the top of the brewery, and ample provision made by having louvre-board windows for ventilation, it takes a long time for the wort to cool down to the required temperature, especially in warm weather, and the large surface exposed to the air offers great liability to infection by microbes from the air. It is now the practice

to cool only to about 140° F., and then complete the cooling by allowing the wort to flow over the refrigerator. Besides cooling, however, the wort becomes aerated, and certain proteid matters are thereby rendered insoluble and are precipitated as a slime in the cooler. Aerating cooled wort does not effect this result at least to the same extent, and wort aerated cold gives a turbid filtrate. Recently the desired result has been attained by spraying the hot wort into a deeper cooler, thus saving considerable space with less liability to infection from the air, while the refrigerator is made to do the principal part of the cooling. The refrigerator consists of a series of horizontal tubes through which cold water passes from the lowest tubes to the highest, the wort meanwhile flowing over the outside in a thin stream until it leaves at a temperature of 60° F. to enter the fermentation tank. The tubes need to be kept scrupulously clean to avoid infection by microbes which may lodge at the angles, more is to be feared from colonies of these which have grown habituated to the wort than to the chance infection from those falling into it from the air during the comparatively short time it is thus exposed.

## Section IV.—FERMENTATION

The cooling of the wort completes the brewing proper, the subsequent operation being the fermentation in which the maltose is converted by the action of the yeast into alcohol and carbon dioxide, and the liquid changed thereby from a sweet wort into an alcoholic fermented beverage, beer. As soon as the wort from the refrigerator, cooled to about 60° F., has started running into the fermentation vessel a quantity of yeast, called the pitching yeast, is added in the proportion of one to three pounds to the barrel of 36 gallons, according to the various conditions of working and the quality of beer to be made. The yeast is first well roused up with a portion of wort in order to secure its uniform distribution. At this point the excise official takes the dips and gravities to determine the duty chargeable.

After a few hours small bubbles of carbon dioxide may be seen rising to the surface of the liquid, and these soon collect into a frothy layer which is fancifully named from its various appearances as *cauliflower head* and *rocky head*, and ultimately becomes more compact and yeasty through which large bubbles of carbon dioxide penetrate and burst, keeping the whole in a state of continuous motion. In about 48 hours the yeast is skimmed off, either with skimmers by hand or mechanically, and rejected, and the operation of skimming is repeated at intervals several times, this yeast being collected in the yeast backs for pitching subsequent charges. When fermentation has proceeded far enough, as shown by the liquid appearing dark and free from suspended yeast when a portion of the yeast head is brushed aside, the last head is allowed to remain and act as a protection against contamination by air-borne microbes, and the whole is allowed to rest

to settle and clear, and then drawn off into casks, or it is run off into a special racking vessel where, after a rest not exceeding 24 hours, it is then filled into casks. The racking vessel affords a convenient means for adding sugar or other *priming* instead of adding it to each cask. In the racking vessel little or no further fermentation occurs. Instead of completing the fermentation in one vessel as in the skimming system several other methods are employed. The first part only of the fermentation may be allowed to take place in the vessel, and then the partly fermented wort is run out, either into rectangular vessels known as *dropping squares* or into a series of large inter-connected casks, which, from their use in the breweries of Burton-on-Trent, are called *Burton unions*. In these the fermentation is completed.

The simplest form of a fermenting tun is a square or round vessel of wood, often lined with copper or aluminium, and provided with an attemperator, a coil of piping through which cold water may be passed to keep down the rise of temperature which takes place owing to the vital activity of the yeast. The rise should not be allowed to exceed about 70° F. The difficulty of producing a good, drinkable beer in hot climates is well known.

Besides the above primary fermentation in which all the readily fermentable sugar has been decomposed, and an equivalent proportion of alcohol formed, there is another slower secondary fermentation which takes place in the cask and serves to keep the beer from becoming flat and undrinkable. This is due to a different yeast, which is either a wild yeast obtaining access during the cooling, or which exists in small proportion in ordinary brewing yeast, and the action of which was overwhelmed during the more vigorous and preponderating activity of the ordinary yeast in the primary fermentation. In the cask this yeast is now free to decompose slowly the malto-dextrins and ferment the resulting maltose into alcohol and carbon dioxide and thus keep the beer sound.

**Yeast.**—Ordinary brewers' yeast, when observed under the microscope, is seen to consist of a number of minute,

slightly ovoid cells, 8 to 9$\mu$ in diameter. Each cell forms a distinct plant, bounded by a transparent cell wall enclosing the protoplasm and other cell contents. In young cells the protoplasm is clear and transparent, but in the older cells appears granular and contains one or more apparently empty spaces, the vacuoles. Many of the cells have smaller ones attached to them so as frequently to form chains. This is the usual mode of reproduction, a protuberance or bud forming on the side of the cell, and after growing sufficiently becoming detached. Another mode of reproduction is by the formation of ascospores, which are produced when young yeast is kept at a suitable temperature, about 25° C., on a moist surface. The protoplasm becomes granular and subdivides to form two, four or eight separate portions which become invested with a membrane and constitute the spores. When the cell bursts each of these spores becomes a new yeast plant and can reproduce by budding as before. This property of forming spores has been utilized by Hansen in differentiating the various yeasts, the shape of the cells alone not affording a sufficient criterion for this purpose, since the form alters considerably under different conditions of cultivation. The importance of distinguishing the various yeasts will be apparent when it is realized that they differ very greatly in fermenting power and in the quantity of alcohol, and the acidity they produce, and also in the amount of nitrogen consumed and of new cells produced, while the flavour of the beer is also largely dependent on the specific character of the yeast used.

**The Chemical Changes during Fermentation.**—Before the maltose present in the wort can be fermented it must be converted into a corresponding amount of dextrose which is effected by the enzyme maltase secreted by yeast. In like manner cane sugar must first be converted into equal molecules of dextrose and levulose by the enzyme invertase, which is also secreted by yeast. It is only the hexoses which are directly fermentable. The conversion of dextrose into alcohol and carbon dioxide according to the equation $C_6H_{12}O_6 = 2C_2H_6O + 2CO_2$ is only the main portion of the

reaction brought about by the enzyme zymase of the yeast, 5 per cent. of the dextrose being converted into glycerine, carbon dioxide and other products, and also utilized for the growth of the yeast and for the production of new cells. The small proportion of higher alcohols and succinic acid always formed during fermentation are derived from the amino-acids present in the wort.

**High and Low Yeasts.**—There are two kinds of yeast in general use in breweries, the one used in England for the fermentation of infusion worts with frequent aeration, and known as high yeast from the temperature, 60° to 70°, at which fermentation is allowed to proceed. It is also called top yeast, since it rises to the surface of the fermenting liquid. The other kind is used with decoction worts on the Continent for the production of lager beer, and is fermented at a lower temperature, 5° to 6° C., hence called low yeast, and from the fact that it settles to the bottom of the fermenting tun, bottom yeast. Both these cultivated yeasts represent types of one species, *Saccharomyces cerivisæ*, and although low yeast may be made to produce a transient top fermentation, this soon ceases, and it does not appear probable that the one kind can be converted into the other at least by the few fermentations of an experiment. There appear to be three sub-varieties of each of these two brewery yeasts with markedly different properties, and it has been suggested that the different flavour of beer made from similar worts may be due to a preponderance of one or other of these forms in the yeast used.

**Pure Cultures.**—A great reform in the fermentation industry was made in 1879, when Hansen, of Copenhagen, introduced yeast produced by culture from a single cell, and was thus able to show that yeast which, when examined under the microscope, was apparently pure, in reality consisted of several types or varieties, to some of which unpleasantness of flavour and turbidity was due. A portion of malt wort mixed with enough gelatine to enable it to set when cold is inoculated with yeast so dilute that when a drop of the wort is spread on a microscope cover glass

only a few separate cells are visible, and these so far apart that after incubation the colonies formed from them would not come into contact with each other. After these have sufficiently developed a portion of each is removed and placed in a flask containing sterilized wort. In a few days fermentation will have taken place, and sufficient yeast will have been produced for the pitching of a larger flask of wort, and by a repetition of the operations, using at each a larger quantity, at length sufficient yeast will be obtained to carry out a fermentation on the brewery scale, and thus enable the quality of the resulting beer to be examined. When a yeast is found which, under the conditions of the brewery gives the quality of beer desired, its use on the large scale conduces to rational working instead of the usual chance results, and diseases of the beer with attendant losses are obviated so long as the yeast is kept pure. It is not necessary to pitch each fermentation with fresh pure culture yeast, since the yeast remains sufficiently pure for practical purposes for some time, and only requires the periodical addition of pure yeast to maintain its satisfactory character. Each brewery requires to select the type of yeast best suited to its conditions to produce the character of beer for which it is known. The use of pure culture yeasts has met with a large measure of success in Continental breweries, but has not proved so advantageous in English breweries, chiefly owing to the necessity for the secondary fermentation in cask essential for these beers. The secondary fermentation is produced by a different yeast from that which gives the primary fermentation, and without it the beer, although sound and of good flavour, is thin and flat. The secondary yeast is present in ordinary pitching yeast. Single yeast cultures, however, are more satisfactory with quick running ales, for which the secondary fermentation is less essential.

**Aeration, Temperature and Duration.**—Yeast cells require oxygen for carrying on their vital activities, and although fermentation will go on in the absence of air, unless the necessary oxygen is supplied the cells soon lose

their fermentative faculty and die. The wort is aerated to begin with during its passage over the refrigerator, and the contents of the fermenting tun are roused from time to time to supply air. Excessive aeration, on the other hand, leads to an unnecessary development of yeast cells at the expense of the sugar, and is attended with a diminution in the yield of alcohol.

The temperature at which fermentation is allowed to proceed varies with the kind of beer made, weaker qualities are usually worked at a temperature not exceeding 70° F., but with stronger beers the temperature may be allowed to rise as high as 75° F., and this is also the case with stout and dark beers. Low temperature conduces to the production of a finer flavour, and more stable beer and more carbon dioxide is retained in solution. The higher temperatures tend to favour the development of bacteria, but this is somewhat counterbalanced by the more vigorous fermentation, and dark malts contain, moreover, products of an antiseptic character. Higher temperatures lead to irregular development of yeast. In the case of bottom yeasts the temperature is from 38° to 48° F., and fermentation is much more sluggish.

When fermentation takes its normal course the yeast is ready for skimming about 48 hours after pitching, and the primary fermentation lasts usually about 8 days, after which some qualities of beer are sent out for consumption immediately they are racked into casks, while others are kept for some months to mature. The bottom fermentation conducted at a lower temperature proceeds more slowly, and is prolonged to 12 or 15 days, and the beer is then kept in casks stored in cellars at a low temperature until filled into delivery casks. It must be quickly consumed after being removed from the protective influence of the cold cellar, as it does not keep so well as the more heavily hopped English beer.

**Density of Wort and Attenuation.**—A quarter of malt, 336 lbs., is usually taken to yield four barrels of 36 gallons each of wort of a specific gravity of 1·055. The density of

the wort is arranged to suit the strength of the beer to be brewed, and is spoken of as the original gravity of the beer. Brewers also describe worts as of so many pounds, this being the excess of weight of the wort over that of a barrel of water, 360 lbs. Thus a wort of a gravity 1·055 would be called one of $55 \times 36 = 19{\cdot}8$ brewers' lbs., the weight of a barrel of such wort being $360 + 19{\cdot}8 = 379{\cdot}8$ lbs.

Yeast cannot readily ferment worts containing 20 per cent. of fermentable sugar, equal to 51 lbs., and its action is inhibited by the alcohol it produces, so that 14 to 15 per cent. of alcohol is the maximum attainable. The gravity of the wort is taken during the course of fermentation after pouring a sample from one vessel to another to disengage the gas, and is found to diminish steadily as sugar is used up and alcohol produced. The final gravity, termed the attenuation, reached may be $1{\cdot}016 = 5{\cdot}76$ lbs. This determination is affected by the presence of the alcohol, albuminoids, caramel, and hops, and gives only the apparent gravity. Yeasts differ considerably in the attenuation they are able to produce.

**Bakers' Yeast.**—Yeast is mixed with the dough in bread making in order that the carbon dioxide and alcohol generated may confer the desired sponginess to the bread when baked. Brewers' yeast formerly in use for this purpose is, owing to its hop flavour, not so suitable as distillery yeast. The manufacture of pressed yeast is carried out in this country and abroad as an adjunct to the distillation of spirit. The well aerated mash of grain is fermented vigorously at a high temperature, and the yeast skimmed off when the production of young cells has ceased. The yeast is separated in filter presses and preserved in tin-foil wrappers.

**Yeast Food Product.**—The high percentage of nutritive material in yeast has led to its use as a food product since dried yeast contains 45 per cent. of albumenoids derived from the protoplasm of the cells. Yeast extract, prepared by heating well-washed beer yeast and evaporating in a vacuum pan to a syrupy consistency, has the appearance

and odour of meat extract, but is somewhat paler in colour. The chief characteristic difference is the absence of the bases creatine and creatinine which are present in meat extracts. Dried yeast forms a good fodder for sheep, but is not so easily digestible by the human subject. It forms a bulky mass of light-brown flakes, and is excellent for dairy cows, giving an increase in milk yield over cotton seed meal. The bitter taste due to hop resin is removed by treatment with alkali carbonate or disguised by addition of fats and starchy meals.

**Diseases of Beer.**—Beer should be brilliantly clear, any cause of dimness or turbidity is to be scrupulously avoided. Bacteria produce not only undesirable alterations in odour and flavour, but often cause a turbidity not removable by finings. It is advisable, therefore, by the strictest attention to cleanliness to avoid contamination by these organisms. Certain species of yeast also may cause turbidity, as was shown by Hansen, but the wild yeasts *Saccharomyces Pastorianus* and *S. ellipsoideus* are only harmful when present in too great a proportion in the pitching yeast. When this is the case it has been suggested to wash the yeast and separate the noxious forms by decanting them off, while the larger and more useful cells remain behind in the sediment. Yeast turbidity is removable by finings. The finings which it is customary to add to beer are made by soaking isinglass in water acidulated with tartaric, acetic, or sulphurous acid, or sour beer, and adding a portion to each cask of beer when it is sent out from the brewery. Ropiness of beer is due to an organism which produces a gum-like body, dextran, and renders the beer so viscous that it may be pulled out in strings. The malady appears to be caused by *Bacillus viscosus*, and is more frequently met with in lightly hopped beers than in those heavily hopped as in pale ales, and is discouraged by increase of acidity. A sarcina form, *Pediococcus viscosus*, has also been shown to cause this trouble, originating in aerial infection of the worts on the refrigerators. Bacteria also secrete diastatic enzymes which may render the higher

malto-dextrins fermentable by yeast, and thus lead to loss of condition by the beer becoming thin in flavour and losing its palate fullness. The acids produced by bacteria render beer sour and undrinkable. *Bacterium aceti* promotes the oxidation of alcohol and forms acetic acid, as is done purposely in the manufacture of vinegar. Others generate traces of butyric and other fatty acids of disagreeable odour. Other bacteria reduce sulphates and sulphites and yield sulphuretted hydrogen. Casks affected in this way are known as stinkers.

**Zymase.**—The enzyme invertase may be extracted from yeast by water, to which it is advisable that chloroform or toluene should be added. The limiting membrane of the yeast cell is sufficiently permeable to allow the invertase to pass out, but better results are obtained if the yeast cells are allowed to disintegrate by autolysis. The enzyme zymase which brings about fermentation cannot be obtained in this manner, for as this enzyme is intra-cellular the cells require to be disintegrated first, as was first shown by Buchner in 1897. He ground yeast thoroughly with sand and diatomite in a mortar, and then subjected the thick dough-like mass to a pressure of 300 atmospheres in a hydraulic press. The diatomite is essential both as a filter and to act as a support for the cells, so that the immense pressure may squeeze out the fluid contained in them. The great surface of the diatomite holds back a very considerable portion of the enzyme, but a further yield may be obtained by rubbing up the press cake with water and pressing again. The pressed juice containing the enzyme forms a clear opalescent liquid which, when added to wort, causes an evolution of carbon dioxide and production of alcohol in the same proportions as when living yeast is used. Succinic acid and glycerine are also formed as by-products. Like living yeast, it shows specific characters in fermenting, also dextrose and lævulose, but not lactose or mannitol. That the action is not due to minute particles of protoplasm is evident, since the fermentation goes on in the presence of chloroform, which prevents the vital

activity of living protoplasm. Moreover, it is still active when filtered through a Chamberland filter. Harden and Young, in the endeavour to effect a more complete separation of colloidable substance, passed the yeast juice through a Martin filter formed by soaking a Chamberland filter in melted gelatine and allowing the latter to set in the pores. Colloidal matter, soluble in water, was retained on the gelatine, but neither this nor the filtrate were capable of inducing fermentation separately, although when mixed they would do so. It was thus shown that two substances take part in producing fermentation. On the filter was the enzyme with other colloidal matter, and the liquid that passed through the filter was the co-enzyme. Further experiments showed that a hexose phosphate is formed as an intermediate compound during the fermentation and again decomposed. The yeast juice suffers a rapid loss of activity on keeping. This has been shown to be due to the presence of a proteo-clastic enzyme which not only dissolves albumin but also the zymase. Enzymes in a dry state are more resistant to heat than in the presence of water, and retain their activity for a more prolonged period. Buchner dried yeast by spreading it in a thin layer on glass plates and drying as quickly as possible in a current of warm air, and subsequently heating to about 70° C. The zymin or permanent yeast described by Albert was made by stirring yeast mixed with alcohol and drying with ether. It forms an impalpable powder in which the more detailed structure of the yeast cell is no longer recognizable, the protoplasm of the cells is dead, but the zymase present is capable of inducing fermentation.

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# PART IV.—WINE

ALCOHOLIC beverages prepared by the fermentation of fruit juices containing more or less sugar are classed as wines, the juice of grapes being understood when the term wine is used without the qualification usual in describing British wines, as Currant wine, Gooseberry wine, Apple wine (cider), etc. These latter are made in countries with a temperate climate from white, red, or black currants, mulberries, gooseberries, elderberries, or raisins. They require the addition of sugar or honey before they are fermented to make up for their deficiency and raise the percentage of alcohol to the requisite amount. Grape wines are variously classified as white or red from their colour, as dry or sweet on the amount of sugar in the finished product, and as still or sparkling according to their content of carbon dioxide. They are given geographical names to indicate their place of origin, since wines from different districts possess special characteristics. Thus we have Burgundy, Port, Sherry, Madeira, Moselle, Champagne, Tokay, Californian, Australian, Cape, etc. These are distinct in colour, flavour, aroma or bouquet, alcoholic strength, and content of sugar. Taste often demands strong wines so that they are sometimes fortified by the addition of spirit. If brandy distilled from wine is used for this purpose the product may still be regarded as genuine, but when the alcohol added is derived from other sources the result is not true wine in the proper sense. The amount of ethyl alcohol in natural wine varies from 6 to 13 per cent., with small quantities of higher alcohols. The acids present are tartaric and malic acids derived from the grape juice and tannic acid from the skins and stalks with carbon

dioxide, and succinic acid produced by fermentation and also a little acetic acid. The free acid present, calculated as tartaric acid, amounts to 0·3 to 0·7 per cent. The salts present are chiefly potassium tartrate and phosphate. The characteristic aroma of wine is due to œnanthic and other esters, but some of the constituents which confer the highly-prized bouquet of certain vintages or the wines of particular districts are still beyond chemical determination; it is dependent on the particular variety of vine, the locality, the season, and is influenced by the yeasts. The colouring matter in wine is extracted from the skins by the combined action of the alcohol and acid during fermentation, when the skins are left in the mash, otherwise the juice from black grapes, fermented without the skins, gives white wine. The browning of wine, which takes place in course of time, and which is known as ageing, is due to the oxidation and precipitation of the colouring matter; this change can be accelerated by the addition of an oxidase.

Few wines are sold without being mixed. The intoxicating properties of wine are found to be increased by mixing them with other wines of a different age and growth. The flavour is at the same time often improved. Thus a thin port is considerably improved by the judicious addition of a similar wine of full body, or by a little Malaga or rich sherry; and an inferior old sherry may be improved by admixture with a small quantity of full-bodied wine of the last vintage. This art of cellar management is a matter of experience and artistic taste, and is employed to so great an extent that probably few wines reach the consumer in the unmixed or natural state.

## SECTION I.—GRAPES AND THE VINE

GRAPES are the fruit of the vine, *Vitis Vinifera*, of which a great number of varieties are cultivated and many others grow wild. It is probable that the cultivated strains are derived from wild varieties indigenous to the countries in which they are grown, as it has been frequently found that vines transplanted from one country to another gradually lose their valuable character and yield grapes which produce inferior wines. Not all grapes are eatable, grapes good for wine are often not suitable for eating, and the best grapes for eating make inferior or bad wine. In the unripe state grapes contain malates and tartrates chiefly of potassium, but as they ripen tartrates preponderate. Although the actual amount of acid increases during ripening the percentage is less due principally to the increase of sugar in the juice. In some vineyards the grapes are allowed to hang until they reach the maximum of sweetness and maturity and are beginning to decay. Thus highly-flavoured sweet wines as Sauterne and Rheingau are produced, the sweetness being due to inositol. In dry and hot climates the grapes are allowed to passulate or wilt and change into raisins while still on the vine. These give sweet thick syrupy wines as Tokay or, mixed with alcohol, Malaga. Red or black grapes are vintaged between maximum volume and passulation, as the latter destroys the blue colouring matter.

**Pressing and Must.**—The grapes are crushed to extract the juice, and the latter is then separated from the skins and stalks in wooden screw-presses. The juice, or must, obtained in this manner when fermented yields white wine. In the case of red wine it is necessary to allow the skins to remain in contact with the fermenting must for a considerable

time for the colouring matter to be extracted, and it is then advisable to remove the stalks before the grapes are pressed, otherwise the wine acquires an astringent, harsh taste due to the tannins derived therefrom, and takes years to mature and become drinkable.

**Constituents of Must.**—The proportion of must obtained from the grapes varies from 60 to over 90 per cent., according as the stalks are present or not. It contains from 50 to 80 per cent. of water, 12 to 35 of sugar (glucose and fructose), 2 to 3 of extractive matters (pectin, gums, albuminoids, and colouring matters), 0·2 to 1·0 of acid, and 0·2 to 0·5 of ash. The ash consists of 60 to 70 per cent. of potash, 3 to 5 of lime, 3 to 5 of magnesia, 3·5 to 5·5 of sulphuric acid, 14 to 17 of phosphoric acid, and smaller quantities of soda, iron, manganese, chlorine, and silicic acid.

The must produced by the first pressing yields the best wine, the richest and ripest grapes being the first to give up their juice, while with increased pressure the must obtained is from juice of grapes less ripe, which are more acid, and contains more tannins from the skins and stalks. The ratio of acid to sugar present in the must has been used as a criterion of the quality of the vintage; high acidity indicating a bad season.

**Marc.**—The residue after pressing, consisting of the skins and stalks, is called the marc, and is sometimes used to make inferior wine by fermenting it after the addition of sugar. This petiotized wine is distilled to obtain brandy with which to fortify other wine, or is used to prepare acetate of copper or to make wine vinegar. It is also utilized as a cattle food and as a fertilizer.

## Section II.—FERMENTATION

The must from the press readily enters into spontaneous fermentation from the development of wine yeasts which are always present on the skins and stalks of the grapes, or which fall into it from the air. These yeasts are varieties of *Saccharomyces ellipsoideus*, and are capable of withstanding a high acidity and alcoholic content. White wines are fermented in barrels with the bung-hole left open to allow of the escape of carbon dioxide. Valuable red wines are fermented in conical wooden vats open above, since the skins or husks require to be stirred up during the fermentation.

Port wine is fermented in shallow receptacles of masonry in which the grapes are trodden and, after fermentation, pressed. Fermentation in Italy, Spain, and the south of France is carried out at 15° to 24° C., and is a top fermentation, but in Germany and with the finer French wines, a bottom fermentation is made at 5° to 12° C. At the higher temperature the fermentation takes from 3 to 8 days, and the wine produced is fiery, high in alcohol, and lacking in delicate aroma. At the lower temperature the first fermentation lasts a few weeks, but yields a wine of delicate flavour. When fermentation is complete, and the wine has become clear, all the yeast and other suspended matters are deposited on the bottom of the cask or vat, constituting the lees. From this the young wine is racked, *i.e.* decanted or siphoned off, and run into a clean cask.

**Slow Fermentation.**—The casks are filled quite full, the bung inserted, and the wine allowed to remain at rest for several weeks to undergo the slow or still fermentation during which colouring matters, albuminoids, and acid

potassium sulphate separate out and form a crust called argol. After this the wine is usually quite clear and bright, and after being racked into fresh casks for storage usually remains so.

**Storage and Treatment.**—Many chemical changes occur during the time of storage whereby the wine becomes matured. Various esters are formed from the acids present and bring out the bouquet or aroma. In consequence of the loss of alcohol by evaporation during storage the contents of the casks diminish in volume, and it is necessary to add more wine from time to time to keep the cask full lest there should be a development of moulds and bacteria which would spoil the wine. White wines do not clear so well or so quickly as red wines, and although frequent rackings tend to help the clarification, this method is generally not sufficient to make white wine quite clear. Recourse is therefore had to finings. Isinglass softened with wine is added and roused up with the wine. Brandied white wines, such as sherry, are fined with white of egg. Coarser kinds with skimmed milk. Spanish earth, kaolin, and gypsum are also used to clear and brighten wines. Pasteurizing wine at 60° C. stops the tendency to acidification and other diseases to which it is liable. Marble was formerly added to acid wines to correct excess of acidity, but Gall's method is preferable. The acid juice is diluted with water, and a quantity of sugar added to bring the sugar content to the normal amount before fermentation. The method also allows of the sour must of bad seasons, which will not ferment of itself, being utilized. The addition of plaster of Paris to must or to the finished wine, technically called plastering, evidently causes the wine to clarify more readily than it otherwise would do, but injurious effects have been attributed to the acid potassium sulphate formed by the reaction with the tartrates, and several countries have fixed a limit of 2 grammes of potassium sulphate to the litre as the maximum allowable.

**Sweet, Dry, and Effervescing Wines.**—Sweet wines are produced either by the incomplete fermentation of the

sugar present in a rich must or by the addition of sugar to the must or wine, or by the addition of alcohol to arrest fermentation before all the sugar is fermented. When all the sugar is exhausted during fermentation dry wine is the result. Effervescing wines are obtained by bottling the wine before fermentation is completed. The carbon dioxide produced is held in solution under pressure, and when the bottle is opened the escaping gas causes the effervescence. If carbon dioxide is introduced into the bottle from an extraneous source, as in making aerated waters, the wine is liable to yield a deposit on keeping.

**Medicated Wines and other Alcoholic Beverages.**—These are wines containing medicinal drugs; the following are given in the British Pharmacopœia, namely Antimonial wine, Colchicum wine, Iron wine, Wine of Iron Citrate, Ipecacuanha wine, Sherry wine, Orange wine and Quinine wine. The basis may be either sherry or orange wine.

A number of wines are sold containing small quantities of meat extract, these are not regarded as medicated wines by the revenue authorities. Saké is a Japanese drink prepared by the fermentation of rice with the mould *Aspergillus oryzæ*; it contains about 17 per cent. of alcohol, and is a light yellow colour.

Toddy is a similar beverage made in India by the fermentation of the sugar in the sap of various palms. Arrack is obtained by distilling toddy; it is exported from Ceylon to the United Kingdom, where it is used as an ingredient in making "punch."

Koumiss, the drink of the Tartars of South-Western Asia, is obtained by the fermentation of mare's milk; in Switzerland an imitation of the Russian koumiss is made from cow's skimmed milk, which is sweetened, and fermented with yeast.

activity of living protoplasm. Moreover, it is still active when filtered through a Chamberland filter. Harden and Young, in the endeavour to effect a more complete separation of colloidable substance, passed the yeast juice through a Martin filter formed by soaking a Chamberland filter in melted gelatine and allowing the latter to set in the pores. Colloidal matter, soluble in water, was retained on the gelatine, but neither this nor the filtrate were capable of inducing fermentation separately, although when mixed they would do so. It was thus shown that two substances take part in producing fermentation. On the filter was the enzyme with other colloidal matter, and the liquid that passed through the filter was the co-enzyme. Further experiments showed that a hexose phosphate is formed as an intermediate compound during the fermentation and again decomposed. The yeast juice suffers a rapid loss of activity on keeping. This has been shown to be due to the presence of a proteo-clastic enzyme which not only dissolves albumin but also the zymase. Enzymes in a dry state are more resistant to heat than in the presence of water, and retain their activity for a more prolonged period. Emil Fischer dried yeast by spreading it in a thin layer on glass plates and drying as quickly as possible in a current of warm air, and subsequently heating to about 70° C. The zymin or permanent yeast described by Albert was made by filtering yeast mixed with alcohol and drying with ether. It forms an impalpable powder in which the more detailed structure of the yeast cell is no longer recognizable, the protoplasm of the cells is dead, but the zymase present is capable of inducing fermentation.

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GRAPES are the fruit of the vine, *Vitis Vinifera*, of which a great number of varieties are cultivated and many others grow wild. It is probable that the cultivated strains are derived from wild varieties indigenous to the countries in which they are grown, as it has been frequently found that vines transplanted from one country to another gradually lose their valuable character and yield grapes which produce inferior wines. Not all grapes are eatable, grapes good for wine are often not suitable for eating, and the best grapes for eating make inferior or bad wine. In the unripe state grapes contain malates and tartrates chiefly of potassium, but as they ripen tartrates preponderate. Although the actual amount of acid increases during ripening the percentage is less due principally to the increase of sugar in the juice. In some vineyards the grapes are allowed to hang until they reach the maximum of sweetness and maturity and are beginning to decay. Thus highly-flavoured sweet wines as Sauterne and Rheingau are produced, the sweetness being due to inositol. In dry and hot climates the grapes are allowed to passulate or wilt and change into raisins while still on the vine. These give sweet thick syrupy wines as Tokay or, mixed with alcohol, Malaga. Red or black grapes are vintaged between maximum volume and passulation, as the latter destroys the blue colouring matter.

**Pressing and Must.**—The grapes are crushed to extract the juice, and the latter is then separated from the skins and stalks in wooden screw-presses. The juice, or must, obtained in this manner when fermented yields white wine. In the case of red wine it is necessary to allow the skins to remain in contact with the fermenting must for a considerable

time for the colouring matter to be extracted, and it is then advisable to remove the stalks before the grapes are pressed, otherwise the wine acquires an astringent, harsh taste due to the tannins derived therefrom, and takes years to mature and become drinkable.

**Constituents of Must.**—The proportion of must obtained from the grapes varies from 60 to over 90 per cent., according as the stalks are present or not. It contains from 50 to 80 per cent. of water, 12 to 35 of sugar (glucose and fructose), 2 to 3 of extractive matters (pectin, gums, albuminoids, and colouring matters), 0·2 to 1·0 of acid, and 0·2 to 0·5 of ash. The ash consists of 60 to 70 per cent. of potash, 3 to 5 of lime, 3 to 5 of magnesia, 3·5 to 5·5 of sulphuric acid, 14 to 17 of phosphoric acid, and smaller quantities of soda, iron, manganese, chlorine, and silicic acid.

The must produced by the first pressing yields the best wine, the richest and ripest grapes being the first to give up their juice, while with increased pressure the must obtained is from juice of grapes less ripe, which are more acid, and contains more tannins from the skins and stalks. The ratio of acid to sugar present in the must has been used as a criterion of the quality of the vintage; high acidity indicating a bad season.

**Marc.**—The residue after pressing, consisting of the skins and stalks, is called the marc, and is sometimes used to make inferior wine by fermenting it after the addition of sugar. This petiotized wine is distilled to obtain brandy with which to fortify other wine, or is used to prepare acetate of copper or to make wine vinegar. It is also utilized as a cattle food and as a fertilizer.

## Section II.—FERMENTATION

The must from the press readily enters into spontaneous fermentation from the development of wine yeasts which are always present on the skins and stalks of the grapes, or which fall into it from the air. These yeasts are varieties of *Saccharomyces ellipsoideus*, and are capable of withstanding a high acidity and alcoholic content. White wines are fermented in barrels with the bung-hole left open to allow of the escape of carbon dioxide. Valuable red wines are fermented in conical wooden vats open above, since the skins or husks require to be stirred up during the fermentation.

Port wine is fermented in shallow receptacles of masonry in which the grapes are trodden and, after fermentation, pressed. Fermentation in Italy, Spain, and the south of France is carried out at 15° to 24° C., and is a top fermentation, but in Germany and with the finer French wines, a bottom fermentation is made at 5° to 12° C. At the higher temperature the fermentation takes from 3 to 8 days, and the wine produced is fiery, high in alcohol, and lacking in delicate aroma. At the lower temperature the first fermentation lasts a few weeks, but yields a wine of delicate flavour. When fermentation is complete, and the wine has become clear, all the yeast and other suspended matters are deposited on the bottom of the cask or vat, constituting the lees. From this the young wine is racked, *i.e.* decanted or siphoned off, and run into a clean cask.

**Slow Fermentation.**—The casks are filled quite full, the bung inserted, and the wine allowed to remain at rest for several weeks to undergo the slow or still fermentation during which colouring matters, albuminoids, and acid

potassium sulphate separate out and form a crust called argol. After this the wine is usually quite clear and bright, and after being racked into fresh casks for storage usually remains so.

**Storage and Treatment.**—Many chemical changes occur during the time of storage whereby the wine becomes matured. Various esters are formed from the acids present and bring out the bouquet or aroma. In consequence of the loss of alcohol by evaporation during storage the contents of the casks diminish in volume, and it is necessary to add more wine from time to time to keep the cask full lest there should be a development of moulds and bacteria which would spoil the wine. White wines do not clear so well or so quickly as red wines, and although frequent rackings tend to help the clarification, this method is generally not sufficient to make white wine quite clear. Recourse is therefore had to finings. Isinglass softened with wine is added and roused up with the wine. Brandied white wines, such as sherry, are fined with white of egg. Coarser kinds with skimmed milk. Spanish earth, kaolin, and gypsum are also used to clear and brighten wines. Pasteurizing wine at 60° C. stops the tendency to acidification and other diseases to which it is liable. Marble was formerly added to acid wines to correct excess of acidity, but Gall's method is preferable. The acid juice is diluted with water, and a quantity of sugar added to bring the sugar content to the normal amount before fermentation. The method also allows of the sour must of bad seasons, which will not ferment of itself, being utilized. The addition of plaster of Paris to must or to the finished wine, technically called plastering, evidently causes the wine to clarify more readily than it otherwise would do, but injurious effects have been attributed to the acid potassium sulphate formed by the reaction with the tartrates, and several countries have fixed a limit of 2 grammes of potassium sulphate to the litre as the maximum allowable.

**Sweet, Dry, and Effervescing Wines.**—Sweet wines are produced either by the incomplete fermentation of the

sugar present in a rich must or by the addition of sugar to the must or wine, or by the addition of alcohol to arrest fermentation before all the sugar is fermented. When all the sugar is exhausted during fermentation dry wine is the result. Effervescing wines are obtained by bottling the wine before fermentation is completed. The carbon dioxide produced is held in solution under pressure, and when the bottle is opened the escaping gas causes the effervescence. If carbon dioxide is introduced into the bottle from an extraneous source, as in making aerated waters, the wine is liable to yield a deposit on keeping.

**Medicated Wines and other Alcoholic Beverages.**—These are wines containing medicinal drugs; the following are given in the British Pharmacopœia, namely Antimonial wine, Colchicum wine, Iron wine, Wine of Iron Citrate, Ipecacuanha wine, Sherry wine, Orange wine and Quinine wine. The basis may be either sherry or orange wine.

A number of wines are sold containing small quantities of meat extract, these are not regarded as medicated wines by the revenue authorities. Saké is a Japanese drink prepared by the fermentation of rice with the mould *Aspergillus oryzæ*; it contains about 17 per cent. of alcohol, and is a light yellow colour.

Toddy is a similar beverage made in India by the fermentation of the sugar in the sap of various palms. Arrack is obtained by distilling toddy; it is exported from Ceylon to the United Kingdom, where it is used as an ingredient in making "punch."

Koumiss, the drink of the Tartars of South-Western Asia, is obtained by the fermentation of mare's milk; in Switzerland an imitation of the Russian koumiss is made from cow's skimmed milk, which is sweetened, and fermented with yeast.

## SECTION III.—TARTAR

**Lees.**—The solid matter deposited in the fermentation vats and casks at the end of the primary fermentation is termed "lees." It consists of impure hydrogen potassium tartrate mixed with yeast and other organic substances, and when mixed with water is extremely difficult to filter unless it has been previously subjected to steam heat for some time to destroy its slimy, colloidal nature. The lees from plastered wines contain calcium tartrate instead of the potassium salt, consequently it is not of much value for the manufacture of tartar.

**Argol.**—Argol is the thin crust which forms a crystalline deposit on the sides of the fermentation vessels, and its 40 to 70 per cent. of tartaric acid is present chiefly as potassium hydrogen tartrate. Its solubility diminishes with the increase of alcohol during fermentation, and the amount of the deposit formed is some indication of the strength of the wine. By extracting argol or lees with hot water and recrystallizing the potassium salt is purified, the operation being aided by the addition of clay and animal charcoal, the latter having been previously freed from its calcium phosphate by treatment with hydrochloric acid. The product is known as tartar and, from the circumstance that a hot saturated solution when suddenly cooled becomes covered with a dense layer of white crystals, it is termed cream of tartar.

**Tartaric Acid.**—Tartaric acid is manufactured from tartar by dissolving it in water and adding whiting chalk until effervescence ceases, the solution being stirred meanwhile and heated by steam. Calcium tartrate and neutral potassium tartrate are thus formed. The potassium tartrate

is then decomposed by boiling with calcium sulphate derived from a previous operation whereby the whole of the tartaric acid is converted into insoluble calcium tartrate and potassium sulphate which remains in solution, and which is run off to be crystallized as a valuable by-product. The calcium tartrate is washed with cold water and decomposed with the calculated amount of sulphuric acid to form calcium sulphate and free tartaric acid which remains in solution. The solution is concentrated in the vacuum pan at 50° to 55° C. and crystallized. In order to obtain crystals of good colour the acid is redissolved, heated with animal charcoal, filtered, and again crystallized; the addition of a little sulphuric acid facilitates crystallization, as the acid is less soluble when sulphuric acid is present. Tartaric acid from its pleasant acid taste is used in effervescing drinks, and in medicine. It is also largely used in the manufacture of dyes, in dyeing, and in calico-printing.

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# PART V.—DISTILLATION

## SECTION I.—GRAIN SPIRIT

STARCH is the source of by far the larger part of sugar from which alcohol is obtained; the starchy material is first ground and treated with water to gelatinize the starch. This forms the "mash," which is then saccharified by means of malt, which is usually germinated barley; the resulting liquid is known as the "wort," and consists chiefly of maltose and dextrins. The wort is then fermented by yeast, which converts the sugar into alcohol, and the resulting liquid is known as the "wash." Finally the wash is distilled to separate the alcohol, which is subsequently "rectified" by further distillation to such a degree of strength and purity as may be desired.

If molasses or beet roots are used as the source of alcohol, malting is unnecessary, a solution of the material is prepared to contain a suitable proportion of sugar and fermented directly.

The following cereals are used in the production of alcohol: barley, rye, maize, millet, oats, wheat, rice, ragi, guinea-corn, and buckwheat.

**Malting and Mashing.**—These starchy substances are generally mixed with two parts of water and steamed under pressure in a conical iron vessel known as a "converter," the temperature being maintained at about 80° C. After steaming for about two hours, a valve in the bottom of the converter is opened, and the charge is passed into a mash tun fitted with rotating stirrers, where the gelatinization finishes as the mass cools down; the cooling being assisted by means of an external water jacket. When the temperature reaches 50° to 55° C. about 4 per cent. of malt is mixed

with the mash, the effect of malting is to develop the enzyme of the barley known as diastase, which converts the starch into sugar.

The barley used to set up artificial germination is steeped in water for 2 to 4 days, during which period it absorbs about one-half of its weight of water and the grain becomes softened; calcium bisulphite is often added to destroy harmful micro-organisms. The steeped barley is then spread out in a level heap upon the malthouse floor, where it commences to germinate, it absorbs oxygen and gives off carbon dioxide, as the growth of the embryo proceeds a small white rootlet appears at the end of the corn; when the growth has proceeded far enough the heap is spread out more thinly to allow the grain to gradually dry. The temperature is not allowed to rise above 15·5° C.

When the drying is complete the product is known as green malt, and it is then removed to the drying kiln, where it is gradually dried more completely, the temperature at the finish reaching about 100° C.

The length of time which is allowed for germination depends upon the use for which the malt is required. For distilling and vinegar making the usual time is 20 days, while the kiln drying occupies 3 or 4 days.

The conversion is complete when the solution no longer gives a blue colour with iodine, the time required varies from 1 to 3 hours; the mash is heated at this stage to about 75° C. to destroy injurious bacteria with which the wort may have become infected. A better method which is now being adopted is to add a small quantity of hydrofluoric acid or ammonium fluoride during fermentation.

This is not harmful to the diastase, while raising the temperature is liable to impair the micro-organism.

After settling the wort is run off and the residual grains extracted again with hot water, the weaker worts being used for mashing the next charge. The temperature is now allowed to drop to 20° C., and the solid matter removed, the solution is then ready for fermentation with yeast, its specific gravity being between 1·030 and 1·055.

**Fermentation.**—The saccharified wort obtained in the manner described is run into fermenting vessels and treated with yeast to start the fermentation; special distilling yeast is used, that is, selected culture yeasts adapted to distilling conditions. If pressed yeast is added about 2 lbs. are employed for every 100 gallons of wort. Usually, however, a preliminary fermentation is made in a good malt wort, this provides a culture of vigorous young yeast cells, and is added to the main wort in the proportion of 4 to 5 per cent.

During the early stages of fermentation the growth of harmful bacteria is checked, in the past lactic acid was used, but now antiseptics, to which the selected yeast has been acclimatized, are employed. The following substances have been used: bismuth nitrate, calcium bisulphite, hydrofluoric acid, ammonium fluoride, the latter being now most in favour.

In the early stages of fermentation there is a rapid development of yeast, for which the best temperature is about 17° to 21° C.; the temperature rises as the operation proceeds, and the main fermentation in which the maltose and dextrose are transformed into alcohol occurs best at 26° to 30° C. At this temperature a secondary reaction also occurs, any dextrins which remain are acted upon by the diastase and gradually converted into fermentable sugars, which are then attacked by the yeast. The chief fermentation is over in about 48 hours, but the wort, now known as wash, is allowed to remain another day at 25° to 26° C. to complete the process.

The wash is now ready for distillation, and contains about 4½ to 7½ per cent. of alcohol, when the original wort before fermentation has a specific gravity of 1·030 to 1·055, as is usual in most distilleries in this country.

The principal changes during fermentation are—

(1) The conversion of maltose into dextrose.

$$\underset{\text{Maltose.}}{C_{12}H_{22}O_{11}} + H_2O = 2\underset{\text{Dextrose.}}{C_6H_{12}O_6}$$

This is brought about by the enzyme maltase contained in ordinary yeast.

(2) The decomposition of the dextrose into alcohol and carbon dioxide.

$$\underset{\text{Dextrose.}}{C_6H_{12}O_6} = 2\underset{\text{Alcohol.}}{C_2H_5OH} + 2CO_2$$

This change is produced by the action of another yeast enzyme, namely zymase.

However, many secondary reactions result according to the nature of the wort and yeast, chief among the by-products as regards quantity are glycerol, fusel oil, succinic acid, other fatty acids, aldehydes, and various esters.

A complete conversion of starch into sugar and alcohol is impracticable, the theoretical quantity would be—

From dextrose, $C_6H_{12}O_6$ .. 51·1 per cent.
,, maltose, sucrose, $C_{12}H_{22}O_{11}$ 53·8 ,,
,, starch, $C_6H_{10}O_5$ .. .. 56·8 ,,

About six-sevenths of the theoretical yield is obtained.

**The Amylo Process of Fermentation.**—Certain moulds secrete diastase and also fermentation enzymes. This property has been utilized in a process of combined saccharification and fermentation, whereby the use of malt is dispensed with in obtaining alcohol from starchy materials. Moulds possessing this property are *Amylomyces Rouxii*, *Mucor β*, *Mucor γ*, and *Rhizopus Delmar*, the last three are now generally used, as they produce less acid than *A. Rouxii*.

The fermentation stage, using moulds is a slow one, and to hasten it yeast is added; advantages claimed for the amylo process are a better yield of alcohol, a purer product, as the conditions of working exclude bacterial contamination, and an economy of malt; a single gram of the mould is sufficient to convert 25 tons of maize. Compared with operations where 10 to 12 per cent. of malt is used, when this one gram of mould is employed, there is a saving of about 3 tons of malt.

In this country a legal obstacle prevents the use of the amylo process; it is a statutory requirement that the specific gravity of distillers' wort before fermentation shall be ascertained by means of the saccharometer, and, of course, this is impossible.

**Distillation.**—To separate the alcohol from the by-products the mixture is distilled. There are two processes in use, the pot still and the patent still.

Where malt whiskeys are produced, and it is the object of the distiller to retain in the product some of the esters, higher alcohols, and other bodies which give the spirit its characteristic flavour, the apparatus employed is the pot still.

**Pot Still.**—This consists of a boiling vessel with a retort head and a worm condenser; or the head may be attached to one or more fractionating vessels similar to Woulfe's bottles, through which the alcoholic vapours pass before reaching the condenser. Usually the pot still is heated by the direct flame of a furnace, though steam is now frequently used as the heating agent.

The spirit produced by the first distillation is always impure, and has a disagreeable odour and taste, and is known as "low wines." On redistillation of the low wines, the first portion of the distillate, known as "fore shots," is contaminated with oily matters; as the distillation proceeds the oily matter diminishes, and where no turbidity is produced on dilution with water the spirit is considered sufficiently pure to be collected as potable alcohol. At the end of the process the spirit becomes oily again, and is known as "tailings." The "fore shots" and "tailings" are together known as "feints," and are redistilled with the succeeding "low wines"; it is usual to have two separate stills for the two distillations.

The residue from the distillation of the wash is termed "pot ale," and from the low wines is called "spent lees." The former is sometimes evaporated and the product used as a fertilizer.

**Patent Stills.**—By far the greater quantity of ordinary alcohol is distilled in a patent still, the invention of Aeneas Coffey. In this process the alcohol is expelled by blowing steam through the wash which is spread in layers over a considerable surface. The wash enters at the top of the still and passes downwards over a series of perforated copper plates in which are small orifices, where the liquid is

met by the uprising steam. The latter bubbling through the perforations heats the wash and carries off the alcohol vapour, so that by the time the wash reaches the lowest plate, it has been deprived of all its alcohol. The alcohol, steam, and volatile impurities pass to the top of the apparatus which is known as the "analyser," they are then led into the bottom of a similar column of perforated plates termed the "rectifier," where a process of gradual cooling takes place by the action of the pipe conveying the cold wash to the analyser. The alcoholic vapours passing upwards through the plates into one cool chamber after another become cooled, and deposit more and more of their water as they near the top, finally condensing as strong alcohol on an imperforated copper sheet, and passing out. The strength of the spirit thus continuously obtained is 94 to 96 per cent. by volume as against 90 to 92 per cent. by the "pot still" method.

Various other "continuous stills" have been patented, but their essential principle is similar to that of Coffey's apparatus.

**Rectifying.**—When alcohol of special purity is required, it is further purified by special rectification. This is usually done by diluting the spirit with water to a strength of about 45 to 50 per cent., and redistilling it. "Intermittent" stills which fractionate the distillate (the impurities being in the first and last runnings), and continuous rectifying stills are used for the purpose.

Purification by treatment with wood charcoal is also employed, the effect is mainly a chemical one due to oxidation. This filtration may either precede or follow a rectification by distillation. The *Spiritus Rectificatus* or Alcohol (90 per cent.) of the British Pharmacopœia is defined as "a mixture of ethyl hydroxide and water containing in 100 parts by volume, 90 parts by volume of ethyl hydroxide, $C_2H_5OH$. Specific gravity 0·8337." It contains 85·68 per cent. by weight of ethyl hydroxide, and 14·32 per cent. by weight of water.

It should be noted that on account of the contraction

which occurs, Alcohol (90 per cent.) would *not* be given by mixing 90 volumes of true absolute alcohol and 10 volumes of water.

**Absolute Alcohol.**—Anhydrous alcohol is prepared by digesting 95 per cent. spirit with about one-fourth or one-fifth of its weight of quicklime for some days, and then distilling off the alcohol slowly from a water bath rejecting the first and last portions; the resulting product is then treated again in the same way.

When strong spirit is not available it may be obtained by distilling weak alcohol first with potassium carbonate and then with fused calcium chloride.

Other substances which have been used as dehydrating agents are barium oxide, calcium carbide, metallic sodium, and metallic calcium, but, on the whole, quicklime appears to be the best dehydrating agent.

Commercial absolute alcohol usually contains water in quantities varying from about 0·5 to 1·5 per cent. The absolute alcohol of the British Pharmacopœia is defined as ethyl hydroxide, $C_2H_5OH$, with not more than 1 per cent. by weight of water. Specific gravity (at 15·5°/15·5°) from 0·794 (equivalent to 99·95 per cent. of ethyl hydroxide by volume and by weight) to 0·7969 (equivalent to 99·4 per cent. of ethyl hydroxide by volume or 99 per cent. by weight). The test prescribed to exclude excess of water is as follows: Anhydrous copper sulphate shaken occasionally during 2 or 3 hours with about 50 times its weight of absolute alcohol does not assume a decidedly blue colour.

**Proof Spirit.**—Proof spirit of the British Pharmacopœia has a specific gravity of 0·920, which corresponds to a strength of about 49 per cent. by weight of alcohol. The term "proof spirit" is very confusing to many people, and might with advantage be abandoned. Of this there is little chance at present, as it is adopted in several Acts of Parliament, and is the scale to which Sykes' hydrometer, used by the Excise, has reference. The Excise formerly tested the strength of spirits by pouring a certain amount on gunpowder. A light was then applied. If the spirit was above a certain

strength "proof" the gunpowder ultimately inflamed, but if weaker the gunpowder was too much moistened by the water to be capable of explosion, and the sample was said to be "under proof." By Act of Parliament proof spirit is now defined to be a liquid of such density that at 51° F. 13 volumes shall weigh the same as 12 volumes of water at the same temperature. The proof spirit thus produced has a specific gravity of 0·91984 at 60°/60° F., and contains 49·28 per cent. of alcohol by weight, and 57·10 per cent. by volume at 60° F.

Spirits weaker than the above are described as being so many degrees under proof, or so much per cent. under proof (U.P.). Thus, by the term 20 per cent. or 20 degrees "under proof" is meant a liquid containing, at 60° F., 80 volumes of proof spirit and 20 of water. Pure water is 100° under proof. On the other hand, liquids stronger than proof spirit are described according to the number of volumes of proof spirit 100 volumes would yield when suitably diluted with water. Thus spirit of 50° O.P. is alcohol of such a strength that 100 volumes at 60° F., when diluted with water to 150 volumes, would be proof spirit. Owing to the contraction which occurs on mixing alcohol with water, the volume of water it would be necessary to add in this instance would be considerably more than 50 measures. Absolute alcohol accordingly is 75·25 O.P., and contains 175·25 per cent. of proof spirit, for 100 volumes when diluted with water would yield 175·25 volumes of spirit at "proof."

## SECTION II.—POTABLE SPIRIT

**Brandy** is an abbreviation of "brandy wine," signifying burnt or distilled wine. It is usually defined as a spirituous liquid distilled from wine matured by age. Real cognac is distilled from grape juice, and is the most sought-after brandy; the grape is white and small in size and is grown on a calcareous soil. A simple pot still (see p. 174) is generally used, and there are usually two distillations: the first corresponds to the "low wines" of the whiskey, it is very impure, and has a disagreeable odour and taste. The second distillation corresponds to the "spirits."

The pot still varies in capacity from about 150 to 200 gallons, is usually enclosed in brickwork and heated by means of a furnace, wood being used as fuel; some stills, however, are provided with steam pipes and jackets.

In a few districts the spirit is produced from one continuous distillation. Obtained in this way it is not considered so fine as that from the pot still, but is of a higher strength and suitable after flavouring for liquors.

The strength of the wine used varies from 4·5 to 9 per cent. by weight of pure alcohol, or approximately from 10 to 20 per cent. of proof spirit; the quantity used in the process is relatively great, the amount of brandy produced being only 10 to 15 per cent.

The finished spirit as run from the still contains about 64 per cent. by weight of alcohol equivalent to a strength of about 25 over proof. When new, brandy is colourless; for sale it is diluted in vats and coloured either by the addition of caramel, or acquires a yellowish-brown colour by storage in oak casks. Genuine brandy has a sweet mellow flavour, whilst inferior and fictitious brandies can usually be recognized by their fiery taste.

The principal constituents of brandy are acetic, butyric, œnanthic, and valerianic esters, acetic acid, volatile oil, tannin, and colouring matter. The flavour is due to the œnanthic ester.

France is the chief brandy-producing country, and the name cognac is defined by French law to be applied only to spirits produced from wine grown in certain specified districts.

British brandy is made by adding flavouring materials such as esters, bitter almonds, spices, etc., to spirits made from grain, and is chiefly used for cooking purposes. In France, "eau-de-vie," which corresponds to the brandy of this country, is applied to spirit made from a variety of fruits. Brandy is also produced in Spain, Egypt, Algeria, South Africa, and Australia. The Egyptian brandies are said to be distilled from currant grape grown in Greece and Asia Minor.

"Marc" brandy is derived from the skins of grapes after the juice has been extracted; it is used for flavouring other spirits.

**Rum.**—The name is probably derived from "rum-bullion," an old Devonshire term for an uproar, the word being subsequently contracted to rum. This beverage is the product of distillation of the sugar cane; the chief source of supply being the West India Islands and British Guiana. In France it is distilled from beetroot.

There are two distinct types of rum in commerce, Jamaica and Demerara (British Guiana), the chief difference being that the former is the result of slow fermentation of wash of high specific gravity, while the latter is the product of wash of low specific gravity and rapid fermentation. The nature of the soil and therefore the products from the sugar cane also affect the quality of the rum.

Jamaica rum wash consists of the following substances mixed with water: (1) molasses; (2) skimmings from the sugar boiler which sour during storage; and (3) dunder, *i.e.* residue which remains in the pot still after the distillation of the rum; this is fermented and then distilled in a pot

still. The slow fermentation allows of the development of bacterial action in addition to the alcoholic fermentation produced by the yeast, and this appears to be an essential feature in producing the quality and flavour which is characteristic of this rum. This flavour is due to esters of acids of high molecular weight resulting from bacterial decomposition of the dead yeast found in the distilling materials. These esters are absent in rum produced without dunder or acid skimmings.

**Demerara Rum.**—In British Guiana the wash used is prepared by diluting molasses with water and rendering the solution slightly acid with dilute sulphuric acid in quantity sufficient to set free the combined organic acids, but not enough to leave any free sulphuric acid. The reason for making the wash acid is to guard against excessive propagation of the butyric and lactic organisms, and to render it more suitable for active alcoholic fermentations. Ammonium sulphate is sometimes added to supply available nitrogenous food for the yeast. Thus in Demerara rum there is a rapid fermentation due to yeast action, and the bacterial organisms are not allowed time to develop appreciably. Both pot stills and continuous stills (see p. 175) are used in distilling Demerara rum, and the proportion of esters produced is low compared with Jamaica rum.

There are three classes of Jamaica rum :—

(1) For home consumption.

(2) For consumption in the United Kingdom.

(3) For export to Europe, chiefly Germany.

Rums of the third class are fermented for 2 to 3 weeks, and the process is conducted in a strongly acid wash; they have an abnormally high proportion of esters, and are mainly exported to Germany, where they are used for making artificial rum by blending with neutral spirit.

Rum contains a high proportion of volatile acids, and owes its flavour mainly to a mixture of esters, chief among them being ethyl butyrate, formate, and acetate; the colouring matter employed consists of caramel. When new, rum has rather a disagreeable odour, but the true

flavour develops by storing in wooden casks, and continues to improve for 8 or 10 years.

The strength of rum imported into the United Kingdom varies from 20 under proof to 50 or more over proof, the average being 35 over proof, equivalent to 77 per cent. of absolute alcohol. The British Customs distinguish between rums from Jamaica, rums from other sugar cane-producing countries, and imitation rums, a term applied to spirits of rum character made chiefly in Germany by the addition of heavily flavoured rums rich in esters, to plain spirit from potatoes, or beet molasses, with the addition of colouring matter.

**Whiskey.**—The early history of whiskey is somewhat uncertain, the term does not seem to have been employed until the latter part of the eighteenth century; its introduction into Europe is due to the Arabs, but in the grape-growing countries the spirit is naturally produced from wine, whilst in Northern Europe grain is used.

The finest whiskey is manufactured in Scotland and Ireland from malt or mixed malt and grain; there are two processes in use, the pot still and the patent still.

In Scotland two distillations only are usually made. First, from the "wash still," from which the whole of the volatile constituents are collected as "low wines." The "low wines" are redistilled from a similar still, the distillate being collected in three fractions, viz.: (1) "fore shots"; (2) clean spirit or whiskey; (3) feints. The fore shots and feints are added to the "low wines" of the next distilling period. The whiskey fraction is generally run off at a strength of 11 to 25 over proof.

In Ireland the pot stills are larger, some having a capacity of 20,000 gallons; three distillations are made, and the fractions are more numerous than in the manufacture of Scotch whiskey; the strength of the "whiskey" fraction being 25 to 30 over proof.

The patent still, the invention of Aeneas Coffey (for description, see p. 174), has been increasingly employed for the manufacture of whiskey, especially in Ireland. The

spirit obtained from the patent still is stronger than that from a simple pot still.

Scotch whiskeys may be divided into the following five principal classes: (1) Highland malts; (2) Lowland malts; (3) Campbeltowns; (4) Islays, all made from malt in pot stills; (5) grains, a name given to patent still. Irish whiskeys have no similar classification. The first four all contain a peaty flavour, peat being used in the curing of the malt.

The primary constituent of whiskey is ethyl alcohol, the characteristic flavour is due to small quantities (rarely exceeding ½ per cent. of the alcohol present) of esters, acids, higher alcohols and aldehydes produced during malting, fermentation, distillation, and maturing. These products are known as "secondary constituents."

The flavour of newly-distilled whiskey is crude and unpleasant, particularly in the case of pot still whiskey, which contains more "secondary products" than the patent still. By storage in wooden casks changes are brought about in the character of the secondary constituents, with the result that the flavour is improved; this process is "maturing." Patent still spirit costs less to manufacture than pot still, and if the two are mixed, a milder, more uniform, and cheaper product is obtained; by mixing immature pot still spirit—which is naturally cheaper than the fully matured article—with patent still whiskey, the unpleasant taste of the former is toned down so that the mixture becomes more palatable. The proportion of pot still whiskey in cheap blends depends on the price; the cheapest may contain as little as 10 per cent.

**Gin.**—This word is a corruption of the word "genièvre," or junever, the French and Dutch equivalents respectively for juniper, which is the essential flavouring ingredient. It is a spirituous liquor, usually manufactured in a patent still, and made from cereals of which maize, malt, and rye are the chief. The principal flavouring ingredients used besides juniper are angelica root, almond cake, cassia buds, creosote, turpentine, and liquorice powder; the gin is

usually rectified twice, and then flavoured by actual distillation with the juniper berries and other aromatics.

The principal varieties are the English "gin," Geneva, Hollands, and Schnapps, the difference being one of flavour, and each manufacturer has his own recipe, which is preserved as a trade secret.

Geneva is made from a mixed wash of malted barley, rye, and maize in equal proportions, the fermented wash being distilled in a pot still and subsequently flavoured.

Hollands is said to be made as follows: a mixture of 2 parts of ground rye, with 1 of ground barley malt is mashed with about 24 gallons of water for each cwt. of mixed meal. When the mashing is complete the specific gravity of the wort is reduced by cold water to between 1033 and 1038.

It is then fermented and distilled; juniper berries and a little salt are added to the first distillation, and the whole is redistilled. The spirit which now passes over is flavoured with essential oils.

Sweetened gin is made by the addition of sugar syrup to plain gin. Plymouth gin is a special variety used extensively in the West of England, the characteristic flavour is said to be due to ether resulting from the addition of a little sulphuric acid.

The adulteration of gin, except by dilution with water, is not common, alkaline carbonates, alum, and salts of zinc and lead have, however, been found. Gin usually contains 38 to 50 per cent. of alcohol by volume, or 66·5 to 87·5 per cent. of proof spirit. In England it may not be sold below 35 under proof unless declared to be diluted.

**Potato Spirit.**—Potatoes contain on the average 20 per cent. of starch, which is used on the Continent for making alcohol; in 1901 Germany had 12·5 per cent. of her arable land planted with potatoes, the yield being 205 bushels of 60 lbs. per acre. In 1908 about 6400 agricultural potato distilleries were in operation in Germany, producing 84 million gallons of alcohol; the production of spirits from other sources in that country being insignificant.

With suitable flavourings it is used for producing artificial whiskey, rum, brandy, and even wines.

**Liqueurs and Cordials.**—These beverages are strongly spirituous, and usually sweetened compounds flavoured with aromatic herbs, essences, or fruit extracts, and often coloured. There is no real difference between them, but the word "liqueur" is usually applied to foreign products, whilst the British preparations are termed "cordials."

The blending of the flavours in high-class preparations is the art of an expert. Hence care is taken to have a highly-rectified neutral alcohol and pure white sugar as the fundamentals, in order that the flavour of the final product may not be impaired.

Grape spirit is used in high-class liqueurs, but the commoner varieties are manufactured from grain spirit.

The flavouring ingredients are treated in one of the following methods :—

(1) An essence is obtained by macerating the ingredients in alcohol, distilling the mixture, and rectifying the distillate.

(2) An alcoholic extract is made by digestion of the ingredients and filtration, without distilling.

(3) For inferior kinds an essence is prepared by simple solution of essential oils in alcohol.

In all three methods the resulting solution is mixed with the required quantity of sugar, syrup, and alcohol, coloured, if necessary, clarified, and filtered.

The recipes for the better liqueurs, such as chartreuse, are, of course, trade secrets.

**Physiological Action of Alcohol.**—From time immemorial the action of alcohol on the human body has been a controversial subject, and upon many points contrary opinions have been held. The Advisory Committee appointed by the Central Control Board (Liquor Traffic) have published a report dealing exhaustively with the subject, and suggesting basis for further research. The following is a summary of the unanimous conclusions of the Committee :—

**Alcohol as a Food.**—Alcohol is completely and rapidly

absorbed from the stomach and intestine, and distributed by the circulating blood to the different organs of the body. The disappearance of alcohol from the body, apart from the small proportion escaping unchanged, is due to its being oxidized. None of the alcohol is known to be converted into any substance which the body can retain. The energy liberated by the combustion of a *moderate* amount of alcohol can be used by the body to its full value.

Alcohol can, within limits, replace an equivalent amount of carbohydrate, or fat, in a diet, and has a similar effect in economizing proteins. Therefore alcohol has a certain food value in a limited sense, namely, as a fuel or supplier of energy, this theoretical value is, in practice, restricted and counterbalanced by its other properties; and in any case it has, from the point of view of its food value alone, no advantages over a foodstuft like sugar.

**Mental Effects.**—On the brain and nervous system alcohol acts as a narcotic.

The successive phases of inebriation cannot be sharply distinguished, yet three main stages can be made out. The first stage shows a weakening of self-control, and a blunting of the power of self-criticism. The second stage is that in which the sense-perception and skilled movements are disturbed, the drinker begins to show a certain clumsiness of behaviour, and emotional instability. In the third stage self-criticism and control are virtually suspended, the emotional tendencies are weakened, and only strong appeals evoke any response, and in their absence the drinker sinks into a heavy sleep.

There appears to be no certain evidence that alcohol acts as a stimulant on the nervous system, and recent experiments support the conclusion that the direct effect of alcohol upon the nervous system is, in all stages and upon all parts of the system, to depress or suspend its function, but when taken in moderate doses, in dilute form, and at sufficient intervals, it has no effect of any serious and practical account.

**Action on the Digestion.**—Under this heading it is

necessary to consider firstly the effect of alcohol on secretion of digestive juices. There is no doubt that the flow of saliva and the gastric juice are increased, but the latter differs from normal gastric juice, inasmuch as it is deficient in pepsin, it follows therefore that this increased amount of gastric juice is of little or no value in the process of digestion. The second factor is the action of alcohol on movements of the digestive organs. So far as evidence goes, there is no reason to believe the gastric movement is increased by alcohol, while some forms of contraction are arrested. This lessened movement of the stomach is produced by many other volatile substances, and may explain the relief of discomfort and colic-like pains which such substances, including alcohol, may afford. The last factor is the effect alcohol produces on the rate of digestion; 1 to 2 per cent. of pure alcohol has little effect upon the activity of the gastric juice, 5 to 10 per cent. slightly retards digestion, while a concentration of more than 10 per cent. very definitely diminishes the rate of digestion.

Thus it has been found that moderate doses of alcohol have no effect, either one way or the other, upon the digestive organs.

**The Action on Respiration and Circulation of the Blood.**—It has been found by recent experiments that alcohol in moderate doses has no effect on the respiration of practical importance. In large doses a paralysis of the respiratory centre is produced. The old theory that alcohol was a respiratory stimulant has thus been disproved. The supposition that alcohol acts as a *direct* stimulant upon the heart, increasing the frequency and power of its beat, has yet to be proved. Most observers, however, agree that an increase in the pulse rate, lasting about half an hour, follows the taking of a moderate dose of alcohol. There is reason to believe that the accelerated effect noted is due to the weakening of the inhibitory centre which restrains the heart beating, and thus a quickening is observed.

When alcohol appears to promote recovery from fainting, it probably acts simply by virtue of its irritant action on

the mucous membrane of the mouth and throat, much in the same way as the fumes of ammonia or ether will act when inhaled. That the effect is a local and indirect one is evident from the fact that it appears almost at once, long before any significant amount of alcohol can have been absorbed and carried to the heart. In more protracted weakness of the heart, the beneficial effect of alcohol can be attributed to its narcotic and sedative action on the nervous centres which affect the action of the heart.

**Alcohol and the Performance of Muscular Acts.**—Experiments were made on the influence of alcohol on simple reflex action, such as the "knee jerk," and the "eye closing," and upon willed movements, such as the ergograph, hill-climbing, and the speed of starting the movement of turning of the eyes toward a fresh object. It is agreed that a single dose of less than 40 cubic centimetres of alcohol, or as much as would be taken in about 2¾ ozs. of whiskey at proof, or in 1¾ pints of beer, in an adult accustomed to moderate use of alcohol, exerts little or no appreciable influence on the performance by him of a muscular act of simple character not demanding precision. On the other hand, the performance of acts requiring skill tends to be temporarily impaired by a dose of even 30 c.c., the effect being shown especially in a diminution of the speed and nicety of the performance. Reliable evidence that alcohol improves, in normal circumstances, the efficient performance of any muscular act, unskilled or skilled, seems at present to be altogether lacking.

**Effect on the Body Temperature.**—Any feeling of warmth experienced after drinking alcohol is explained by the fact that alcohol causes a slight paralysis of the nerves which control the size of the blood vessels, thus allowing them to distend a little. More blood, therefore, reaches the surface of the body and warms the skin, and the ends of the sensory nerves in the skin, and thus convey to the brain a sensation of warmth. But the sensation is illusory; the body, as a whole, has not really been made warmer, in fact, the temperature has actually been lowered.

It may be stated that under the normal conditions of

life the effect of a moderate dose of alcohol on the body temperature has no importance. In circumstances of brief exposure to cold the discomfort due to a chilling of the face and extremities may be mitigated by the use of alcohol, but the use of it during, or as a preliminary to, prolonged or severe exposure to cold, is to be condemned.

**Poison Action.**—It is stated that ½ pint of absolute alcohol or nearly a pint and a half of proof spirit is a fatal dose for an adult; recent experiments go to show that dilution does not appreciably affect the rate of absorption, and consequently the concentration of the drug in the blood.

There is an important distinction between the effects of a single excessive dose of alcohol, and those produced by immoderate quantities frequently repeated, even though these quantities may be, and usually are, smaller than the single dose which gives rise to intoxication. In the latter case, the drinker returns to his normal state after the alcohol has been eliminated from the body. It is otherwise with chronic alcoholism.

The symptoms of drunkenness are due to the immediate action of alcohol in the body, the effect of which is mainly apparent in disturbance of the functions of the brain. There are two essential factors in the causation of chronic alcoholism. First, the drug must be taken in sufficient quantity to exercise an injurious action on the tissues; and, second, the action must be more or less continuous. The regular use of alcoholic beverages does not induce chronic poisoning so long as only moderate doses are drunk. Chronic alcoholism produces a change in the tissues which may be of a permanent character, and this change is not limited to the nervous centre, but may affect in one way or another most of the organs of the body. Largely they are brought about by its detrimental action on the lining membrane of the stomach and bowel, causing chronic catarrh and failure in the action of the digestive juices, and so promoting the absorption from the alimentary canal of poisonous microbial toxins.

Some affection of mind is usually present in chronic

alcoholism, but it is usually moderate in degree. In delirium tremens more intense, but transitory disorders occur. It appears that in chronic intoxication the nervous system is impaired in ways which affect its healthy functioning; it also contributes towards the development of Bright's disease of the kidneys, and lowers the defences of the body against microbial invasion. There is no evidence to support the popular belief which attributes to alcohol a protective value in cases of exposure to infection.

Recent observations and experiments appear to indicate that parental alcoholism may have a seriously detrimental influence on the stock, but in view of the extreme importance of this conclusion, it is considered well to suspend judgment until the subject has been further investigated.

**Longevity.**—It is found necessary in drawing conclusions between the consumption of alcoholic beverages and longevity to be cautious, as the scientific facts are very limited; but, on the whole, the evidence is in favour of the total abstainer, their death-rate is lower, and expectation of life longer. It should be added, however, that it does not follow from these conclusions that a consistently moderate use of alcoholic beverages shortens life. Summing up, it may be stated that the temperate consumption of alcoholic liquors is physiologically harmless; on the other hand, it is certainly true that alcoholic beverages are in no way necessary for healthy life.

## SECTION III.—INDUSTRIAL ALCOHOL

**Industrial Alcohol.**—Alcohol is also manufactured for commercial use, and during the war, when large quantities were required, some of the whiskey and gin distilleries were commandeered by the Government to add to the sources of supply. Spirit used for heating or as a solvent in chemical industry was to be manufactured under the same conditions of excise control as that used for potable purposes, but it is obvious that questions of flavouring, ageing, and so forth, which give the potable spirits their special characteristics need not be considered when the alcohol is not to be used for food.

**Molasses.**—Both beet and cane sugar molasses are used for the manufacture of commercial alcohol; the former are sterilized by slightly acidifying the diluted molasses and boiling the solution.

Cane sugar molasses as used in this country are simply dissolved in water to make a wort of specific gravity about 1030 to 1040; in the tropics juice expressed from the sugar cane and containing 14 per cent. of sugar is fermented directly. A wild yeast is found on the surface of the cane, and this sets up an active spontaneous fermentation when the juice is kept at a temperature of 30° to 35°.

**Potatoes.**—Potatoes are rarely used in this country for making alcohol, but on the Continent they are largely employed. They are generally steamed under pressure to gelatinize the starch as a preliminary to the mashing operation.

The steaming is usually effected in a conical iron vessel known as a "converter," which is commonly of a size to take a charge of 2 or 3 tons of potatoes. In this vessel the potatoes are subjected to the action of steam at a pressure of 3 atmospheres during 45 minutes or so, the temperature rising to about 135° C. On opening the discharge valve at the bottom of the converter when the steaming is finished the softened mass of potatoes is forced out by the pressure

and passes through a grid or cutting arrangement which helps to complete the pulping of the mass. The latter then goes to a mash tun, where it is cooled by water flowing through coils in the tun, or, in some firms, by means of an external water-jacket.

The starch is converted into sugar, as already described, by the enzyme diastase, supplied by malt, or by the moulds in the "amylo" process. The proper quantity of malt employed amounts to 1½ to 3 per cent. of the weight of potatoes used.

**Alcohol from Sugar Beet.**—There are two chief methods. In one process the beet are sliced and extracted with water in a series of vessels which are so arranged that the water issuing from the bottom of one vessel passes into the top of the next, percolates downwards through the packing of beet slices, and extracts the sugar as it goes; by the time the end vessel of the series is reached the juice has become sufficiently rich in sugar to be ready for fermentation. To minimize the action of harmful bacteria during fermentation the juice is slightly acidified with sulphuric acid. In the second method the beet are topped, the roots and tops being stored separately, and the tops worked off first, as they lose sugar during storage. The extract is made by steaming under a pressure of 2 atmospheres maintained for about 1½ hours in a conical converter, all the sugar being then dissolved, and the beet tissues reduced to a pulp. The chief countries which use beet as a source of alcohol are France, Austria-Hungary, and Germany.

**Alcohol Denaturants.**—The object of denaturing is to prevent duty-free spirit from being put to uses other than those authorized. It should fulfil the following conditions:—

(1) It should impart a taste or smell sufficiently disagreeable to prevent the alcohol being drunk, even after dilution, sweetening, or flavouring.

(2) It should not be capable of being eliminated easily by filtration, distillation, or any other process which can be readily applied or which is ordinarily used in manufacturing operations.

(3) It should be capable of being easily and certainly

detected even when present only in minute quantities.

(4) It should mix readily with the alcohol and produce a mixture of essentially the same properties as undenatured alcohol, and capable of being used in the same way as undenatured alcohol in manufacturing processes.

(5) Its cost should not materially add to the price of denatured spirit as compared with that of ordinary alcohol.

No single substance has yet been found which fulfils all the above conditions. Crude methyl alcohol or wood naphtha fulfils (2), (3), and (4), but as regards the first condition, when mixed with ethyl alcohol to the extent of 10 per cent. does not prevent the alcohol being drunk.

Wood naphtha does not quite satisfactorily fulfil the fifth condition, inasmuch as it renders the denatured alcohol slightly more costly than pure alcohol.

To render the spirit nauseating some other denaturant, such as mineral naphtha or pyridin are employed, where the conditions under which the alcohol is to be used do not admit of strict Revenue supervision. Thus we get two classes of denatured alcohol :—

(1) Denatured for general household use and minor manufacturing purposes.

(2) Denatured for manufacturing purposes on a large scale.

In this country the composition of (1) is as follows :—

90 parts ordinary alcohol.
10 ,, wood naphtha.
$\frac{1}{8}$ per cent. of mineral naphtha and sufficient dye to give it a violet tint.

Practically the same mixture is used in France, U.S.A., Canada, and other British Colonies. Such a mixture is undrinkable even when sweetened or flavoured.

(2) Denatured alcohol for manufacturing purposes contains :—

95 parts ordinary alcohol.
5 ,, wood naphtha.

The mixture is not undrinkable when diluted, sweetened, and flavoured, and greater precautions have to be taken by the Revenue authorities to prevent its illegal use. Application for its use must be made to the Board of Customs and Excise stating clearly the purpose for which it is to be used. Revenue officials must be allowed to visit all parts of the factory, and take samples of the spirits and products at any stage of the manufacture.

In Australia a special committee have reported in favour of the use of the distillate obtained from coal-tar oil at a temperature of from 170° to 230° C. as a suitable denaturant. Experiments with this material are now being made at the Government laboratory.

**Alcohol and Benzol as a Fuel.**—Quite recently the London General Omnibus Company have been experimenting with a mixture of alcohol and benzol as a fuel for the internal combustion engine. Neither burns so rapidly as petrol, and the use of the mixture necessitates constructional alteration in the engine, with special reference to the compression, carburettor, and heating of the induction pipes. A 50 per cent. mixture was found to be the most satisfactory, its efficiency as compared with petrol was as 12·5 is to 15.

As to cost, this may be represented by the number of B.T.U.s per penny produced by petrol—6050; and by the 50 per cent. mixture—5000. The cost per mile showed a difference in favour of the former of one-fifth of a penny. The mixture was found to be 12 per cent. more economical than petrol; the difference is 8 per cent. only in cost, petrol being the cheaper to use if the price per gallon of the two is the same. It has also been found that the thermal efficiency increases with the increased proportions of alcohol for the same compression; that a higher proportion of alcohol means also that the compression can be raised, with a consequent rise in the thermal efficiency. Tests with a mixture of alcohol and ether are now being carried out.

## Section IV.—SYNTHETIC ALCOHOL

The rapidly increasing use of alcohol in the arts and sciences as a solvent and for fuel, and the extensive development of the motor industry, has brought about such a demand for alcohol, that it is feared present supplies will be far from sufficient to meet future requirements. It, therefore, becomes necessary either to increase the production of methyl or ethyl alcohol by increasing the number of present plants if the raw materials required are available, or, if these are only obtainable in limited quantities, to discover other methods of manufacture from other more abundant sources.

Evidently the first part of the question resolves itself into. What are the products which are available in unlimited quantities? and ordinary experience tells us that we must depend on Nature to obtain such materials. In order of their importance they may be enumerated as follows :—

(1) *Wood.*
  (*a*) The ordinary wood not suitable for trade purposes.
  (*b*) Sawdust—wood pulp.

(2) *Starch.*
  (*a*) Cereals.
  (*b*) Potatoes.

(3) *Peat.*

(4) *Carbon.*—Easily obtained by the destructive combustion of any organic matter.

The work which has been done on each of this class of materials for alcohol production will, therefore, be briefly outlined.

(1) **Wood.**—There are several ways in which wood may be treated so as to render a combustible liquid fuel.

(*a*) *Destructive Distillation.*—Here wood logs, shavings, and other refuse is burnt in a closed vessel and the products are condensed and collected. The process is very ancient. Various forms of stills and many modifications of distillation are employed, but the products obtained are as follows :—

| Products. | Per 4 cords. | | |
|---|---|---|---|
| | (High resin.) Rich wood. 16,600 lbs. | (Low resin.) Lean wood. 16,000 lbs. | Hardwood. 14,000 lbs. |
| Charcoal .. .. .. .. | 4284 lbs. | 4400 lbs. | 3600 lbs. |
| Refined turpentine .. .. .. | 41·5 gals. | 20·75 gals. | — |
| Pine oil .. .. .. .. | 11·6 „ | 4·2 „ | — |
| Resin spirit .. .. .. .. | 21·5 „ | 12·0 „ | — |
| Resin oil .. .. .. .. | 141·0 „ | 64·75 „ | — |
| Creosote oil .. .. .. .. | 46·5 „ | 21·0 „ | 20 gals. |
| Acetate of lime, 80 per cent. .. | 300·0 lbs. | 350·0 lbs. | 852 lbs. |
| Wood alcohol .. .. .. | 6 gals. | 9·6 gals. | 37·5 gals. |
| Pitch .. .. .. .. .. | 1400 lbs. | 625 lbs. | 720 lbs. |

It is evident that the amount of alcohol (wood spirit) obtained is far too little to enable the wood to be treated by this process economically—at least for the production of alcohol.

(*b*) *Conversion into Sugar followed by Alcoholic Fermentation.*—Wood cellulose may be converted into reducing and fermenting sugars by the process of hydrolysis, and this can be accelerated by the presence of catalytic agents such as acid. Concentrated $H_2SO_4$, $H_2SO_3$, HCl—all have been used. The sugars by fermentation with yeast yield alcohol.

(i.) *The Ewen-Tomlinson Process.*—This consists in treating wood pulp with dilute $H_2SO_4$ and steam under pressure. 25 to 28 per cent. of the wood is rendered soluble, and of this 80 per cent. is fermentable sugar. A yield of 20 to 22 per cent. of saccharoses corresponding to 10 to 11 per cent. of ethyl alcohol, or 35 gallons of 95 per cent. spirit per ton of dry wood is thus obtained.

The fermentation is carried out in accordance with the standard practice of four-day fermentation period.

(ii.) *Waste Sulphite Liquor.*—Here bone dry wood pulp is

heated with calcium bisulphite solution (2·5 to 3·0 per cent. free $SO_2$) under pressure to 140° C. for 10 to 12 minutes, or 15 to 20 hours at 135° C., 14 per cent. fermentable sugars are obtained. The solution is neutralized with lime or calcium carbonate, allowed to settle, cooled, and run into fermentation vats. High resistant yeast—type XII.—is used to effect the fermentation.

(2) **Starch.**—Cereals such as barley, oats, maize, and even potatoes have been used for the production of alcohol for some time past. The grains (*e.g.* barley) are first allowed to germinate, whereby the ferment diastase is produced. The ferment converts the starch of the cereals into maltose, and this by the ordinary yeast fermentation produces alcohol.

Cereals and potatoes are, however, a staple article of food, and unless cultivation is enormously increased, this source of alcohol production will remain too expensive for the production of industrial spirit.

(3) **Peat.**—Peat may be carbonized in very much the same way as wood, and yields the following quantities of products:—

| Products. Per ton. | Oldenburg. 31% $H_2O$, ·7% N. | Scotch. 16% $H_2O$, 2·1% N. |
|---|---|---|
| Oils .. .. .. .. .. | 54·0 lbs. | 50·0 lbs. |
| Paraffin wax .. .. .. .. | 6·0 „ | Not determined |
| Phenols .. .. .. .. | 26·0 „ | 28·0 lbs. |
| Methyl alcohol .. .. .. | 6·8 „ | 5·1 „ |
| Ammonium sulphate .. .. | 6·2 „ | 30·2 „ |
| Calcium acetate .. .. .. | 10·0 „ | 8·0 „ |
| Pitch .. .. .. .. .. | 4·0 „ | 3·5 „ |

(4) **Carbon.**—The direct production of alcohol from carbon, hydrogen, and oxygen is attractive, as these primary materials are available in unlimited quantities. A successful process of manufacture from the elements would of necessity oust all the other processes, as the latter must depend on various natural uncontrolled forces. The processes attempted are as follows:—

(i.) *From Acetylene.*—This substance is a gas, and can be obtained either by passing hydrogen through an electric arc struck between graphite poles, or technically from calcium carbide obtained in the electric furnace by the action of lime on carbon.

$$CaO+3C=CaC_2+CO$$

Calcium carbide on interaction with water produces acetylene.

$$CaC_2+2H_2O=Ca(OH)_2+C_2H_2$$

Acetylene by catalytic hydrogenation is converted into acetaldehyde.

$$CH=CH+H_2O\rightarrow CH_3CHO$$

The catalytic agents used are sulphuric acid (20 to 25 per cent.). Phosphoric acid 30 to 35 per cent. or solutions of organic sulphonic acids, glacial acetic acid with a mercury salt, ferric mercuric sulphate, etc.

The acetaldehyde by catalytic hydrogenation (Sabatier's process) over reduced nickel is converted into ethyl alcohol at 140° C.

$$CH_3.CHO+H_2=CH_3.CH_2OH$$

The latter process requires careful manipulation in order to prevent the reverse reaction taking place. The process has been successful, and the Lonza Electricity Works at Visp will shortly be able to satisfy the total alcohol consumption of Switzerland.

Another process of hydrogenation, started in France in 1917, consists of electrolysing the aldehyde or paraldehyde in an acid medium, the aldehyde being obtained from acetylene either previously by any process or in the electrolytic apparatus concurrently with the hydrogenation. Sulphuric acid of 5 to 10 per cent. strength is usually employed, but other acid electrolytes may also be used. The aldehyde or paraldehyde is added to the cathode chamber in two stages, first up to 10 per cent., and then up to 30 per cent. The temperature is kept at about 40° C., which is maintained by the heat of hydrogenation. A porous diaphragm is used in the electrolytic cell to prevent

the diffusing of the alcohol and the aldehyde towards the anode. The cathode may be of lead, lead alloy, or mercury. The anode is an inert material, such as platinum, lead, or iron oxide; the current should be 2 to 3 amperes per square decimetre of the cathode. Alcohol may be obtained directly from acetylene by passing it through the cathode chamber, provided the electrolyte contains a mercury salt.

(ii.) *From Ethylene.*—This is a constituent of coal gas, but no technical synthetic process for producing it has yet been devised. The removal of ethylene from coal gas has been attempted, but trouble has been found in the subsequent process when the ethylene has to be converted into alcohol. The conversion of acetylene into ethylene by the addition of hydrogen is theoretically possible, but this hydrogenation has not been successfully accomplished.

Ethylene can be converted by simple hydration into alcohol. The process is catalytic and occurs by way of the intermediate ethyl sulphuric acid, $C_2H_5HSO_4$, and given a large supply of ethylene, the conversion into alcohol does not offer great difficulties. Its recovery from coal gas in this way is therefore theoretically possible, and Bury, at Skinningrove Coke Oven Works, has obtained 1·6 gallons per ton of coal carbonized from runs of 5800 tons of coal carbonized per week (*Chemical Age*, 1919, 1, 714).

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# PART VI.—VINEGAR

## SECTION I.—PREPARATION OF THE WORT

IN all the processes for the manufacture of vinegar advantage is taken of the oxidizing action of the vinegar fungus; the two chief are *Mycoderma aceti*, also known as *Mycoderma vini*, and the *Bacterium xylinum*. These ferments are capable of converting fermented alcoholic liquors into acetic acid; they are white gelatinous nitrogenous plants requiring for their growth proteid substances and mineral salts which are always present in wines and other alcoholic liquids. The fermentation is due to enzymes in the plant cell.

Pasteur examined the *Mycoderma aceti*, cultivated it in synthetic media, and proved that it acted as a carrier of oxygen from the air to the alcohol, and that it was capable when no more alcohol was available of oxidizing acetic acid to carbon dioxide and water. The highest concentration of acetic acid that can be obtained by the use of this organism is 6·6 per cent.

*Bacterium xylinum*, investigated by Brown, occurs in the vinegar plant, or mother of vinegar, in which it is associated with a yeast. The mixed growth is capable of converting cane sugar solution into vinegar, the sugar, which is itself not attacked by the bacillus, being hydrolysed and fermented by the yeast, with production of alcohol, which is then oxidized by the organism. The chemical actions of this organism were found by Brown to agree in general with those of *B. aceti*.

A large number of organisms have been found which have a similar action on alcohol, and which include those found in vinegar breweries. They are not very clearly characterized. Further information can be found in various

papers in *Centr. Bakt. Par. Abt.*, ii. 1905–6; and *Garungs-bacteriologisches Praktikum*, Berlin, 1909.

**Quick Vinegar Process.**—This method is applicable to any alcoholic liquid. It differs from other processes in causing the liquid to expose a very large surface to the action of the air, 1 gallon being sometimes exposed over an area of 100 square yards. The vat is usually from 6 to 12 feet high, and has a false perforated bottom, upon which is placed a quantity of thoroughly washed beechwood shavings, nearly filling the vat. Air holes are bored in the vat about 18 inches from the bottom; the top of the tun is a perforated disc, through which twists of cotton or string are passed of sufficient length to touch the shavings. The vinegar is drawn off by means of a pipe, when it rises to a height of about 15 inches. Before commencing the fermentation it is necessary to sour the shavings; the composition of this fluid varies considerably; any alcohol liquid may be used, but the presence of a trace of tarry matter prevents the action.

According to Wagner, the following mixture is generally used:—

4½ gallons (20 litres) brandy.
9 ,, vinegar (42·5 per cent. by weight).
27 ,, water.

To which is added a mixture of bran and rye to promote the growth of the vinegar fungus.

Another standard liquor is:—

50 gallons brandy or whiskey (52 per cent. by weight).
37 ,, beer or malt wort.
3 to 4 volumes of soft water.

Molasses or honey in the proportion of 2 lbs. to 50 gallons is added to produce a more finely-coloured vinegar. The liquid to be fermented is poured over the upper disc and trickles slowly down the twisted threads, and over the shavings, becoming oxidized by the action of the air which

is drawn through the air holes on account of the rise in temperature. The acetified liquid collects beneath the false bottom; it is drawn off and usually passed through the same vat 3 or 4 times before the oxidation is completed. The strongest vinegar that can be obtained by this method contains about 10 per cent. acetic acid.

**Acetates.**—The following are the most important salts prepared from pyroligneous acid, and vinegar.

*Aluminium acetate*, known as "red liquor," or "mordant rouge," is used in dyeing for the production of red colour. One method of preparation is to add a solution of alum to acetate of lime liquor. The lime is precipitated as sulphate, its place being taken by the aluminium forming the acetate.

*Calcium acetate* is prepared by neutralizing acetic acid, or pyroligneous acid, with chalk. It is used as the source of other acetates, and acetic acid: it is also employed in calico printing.

*Copper acetate.*—This occurs as the normal salt, and in two basic forms, as blue verdigris and green verdigris, the colour and composition changing according to the mode of preparation.

Copper acetates are used as pigments in oil and water colours.

*Ferric acetate* is prepared by the addition of calcium or lead acetate to ferric sulphate. It is used as a mordant, and in alcoholic solution as a medicine.

*Ferrous acetate* is prepared by the action of pyroligneous acid on iron turnings, or nails at a temperature of 65° C. It is known as black liquor, and is used in calico printing, dyeing, and colouring furs, leather, wood, etc., black.

*Lead acetate* is produced by the action of acetic or pyroligneous acid on litharge; two basic acetates are also known. It is used as a mordant, pigment, and in medicine.

*Magnesium acetate* as a basic salt is known in commerce as "Sinodor."

*Sodium acetate* is used as a preservative, and as a source for the preparation of acetic acid. *Potassium acetate* occurs in many plants.

## Section II.—ACETIFICATION

**Malt Vinegar.**—This is prepared from an infusion of malt which has first been fermented to produce alcohol. Six bushels of crushed malt are extracted 3 times with water in a circular mash tun supplied with a central stirrer, the first extraction with water at 72° C., the second at a higher temperature, and the third with boiling water. The extracts, which together should not exceed 100 gallons, are passed into a large cast-iron tank, cooled by refrigerators to 24° C., and then poured into a large circular vessel, mixed with 3 or 4 gallons of good yeast, and fermented briskly for about 40 hours. The extract is filtered and stored in casks for some months, whereby the extractive matters which would produce putrefaction are deposited. The "wash" may be treated by the quick vinegar process, or acetified in large casks lying on their sides in a temperature of about 24° C. The bung holes of the casks are open, and at each end near the top an opening is made for the circulation of air. When conducted in the open air the process is known as "fielding." From 8 to 20 rows of casks constitute a vinegar field. The operation is commenced in the spring and completed in about 3 months.

Malt vinegar is a brown liquid of a characteristic odour, due to the presence of esters. It has an agreeable acid taste due to acetic acid. It usually contains alcohol, gum, sugar, and extractive matter, acetates, chlorides, free and combined sulphuric acid, and on evaporation and ignition leaves a residue containing much phosphate.

**Wine Vinegar.**—The following is the plan of making wine vinegar as practised in the district of Orleans. Wines

which have become sour are generally used, if they contain above 10 per cent. alcohol they are suitably diluted with weaker wines. The wines for acetification are kept in casks with beech shavings to which the lees adhere, the wine thus clarified is drawn off to make vinegar. A certain amount of extractive matter is, however, necessary for the proper growth of the plant. The Orleans casks contain nearly 400 pints, and are placed in 3 rows one over another, the upper ones having an aperture of 2 inches in diameter kept always open. 100 pints of good vinegar, boiling hot, are first poured into each cask and left there for 8 days, 10 pints of wine are mixed in every 8 days, until the vessels are full. The most favourable temperature is between 25° and 30° C., and is kept up when necessary with a stove.

The casks are never more than half emptied, but are successively filled up again to acetify new portions of wine. In order to judge if the fermentation is completed at the end of the usual time, which is from 8 to 30 days, according to the time of year, the workman plunges a spatula into the liquid. If reddish froth adheres more wine is added, and the temperature raised. A white froth indicates the completion of the process.

The casks become foul after 6 years. The deposit of argol, yeast, and sediment is then removed, and they are cleansed and recharged with hot vinegar, as in the case of new casks. Good casks will often last 25 years.

Wine vinegar is of two kinds, white or red, according as it is prepared from white or red wine; the former is considered the finer. In addition to the substances usually met with in malt vinegar, it contains potassium tartrate, by which it can always be recognized.

**Vinegar from Alcohol.**—Three million gallons of alcohol are used yearly in Germany for vinegar making. Pure alcohol does not suffice as a medium in which acetic fermentation can take place, since nitrogenous substances and salts are necessary for the growth of the organism. Cane sugar or glucose is usually added in the proportion of 1 to 3 kilos. per 100 litres of pure alcohol, while the nitrogenous needs

are met with inorganic salts, chiefly acid phosphates of ammonium, sodium, and potassium, about 50 to 150 grams are used per 100 litres of pure alcohol. The acetification vessels are cylindrical in form, with a false perforated bottom upon which are beech shavings; in the sides are air holes for oxidation.

The alcohol is diluted to a strength of 6 to 10 per cent. mixed with the required nutrient materials and delivered on top of the shavings; when starting with a newly-packed vessel a pure culture of the bacterium is mixed with the first charge of alcohol; subsequently some of the acetified product is used for mixing with the charges. The temperature of the vessel is kept at 25° to 35° by controlling the admission of air and of the alcoholic charge. The resulting vinegar is collected in the bottom of the vessel, and contains from 4 to 6 per cent. acetic acid. Double or triple strength products can, however, be produced by mixing ordinary vinegar with fresh alcohol and treating with suitable organisms in other vessels; up to 12 per cent. of acid can thus be obtained.

In addition to wine, malt, and alcohol, vinegar is prepared from cider, perry, beer, glucose, and skim milk.

An artificial vinegar is made by mixing acetic acid with water and caramel, and acetic esters to produce the proper colour, odour, and taste. This vinegar differs from genuine kinds by the absence in the evaporated residue of phosphoric, tartaric, or malic acid.

The vinegar of the B. P. has a specific gravity of 1·017 to 1·019, and the minimum of acetic allowed is 6 per cent. Genuine vinegar seldom falls below 5 per cent., and should be condemned as adulterated with water when the amount is as low as 3 per cent.

The chief adulterants are mineral acids, flavouring agents such as cayenne and ginger, and metals derived from the vessels used.

Vinegar manufactured in Germany from denatured brandy contains a certain quantity of pyridine which can be estimated (*see Zeitsch. Untersuch. Nahr. Genussm.*, 1911, 21, 655, 658).

## Section III.—ACETIC ACID

**Acetic Acid, $CH_3COOH$.**—Acetic acid occurs in nature in the juices of many plants, especially trees, either as free acid, or generally as the calcium or potassium salt; and as organic acetates in the oils from many seeds. It is also found in certain animal secretions.

**Formation.**—The greater part of acetic acid used in commerce is obtained by destructive distillation of wood. Hardwood, such as oak, birch, beech, maple, and elm give the best yields of acid and wood alcohol. A full description is given in H. Taylor's book on "Fuel Production and Utilization" of this series, from which the following extracts are taken :—

There are three processes in use :—

(*a*) Kilns of the beehive type with a capacity of 50 to 75 cords, now almost obsolete, owing to low yields of charcoal and loss of by-products.

(*b*) Discontinuous retort plants :—

(1) Small retorts holding from 1 to 5 tons of bulk wood;

(2) Large retorts, or ovens, the charge being 10 to 15 tons in bulk form.

(*c*) Continuous mechanical plants; carbonizing chippings or shavings only.

The retorts are usually steel-plate cylinders which can be revolved to expose new surface to the action of the fire; others are rectangular steel ovens of large capacity, into which the wood may be charged on trucks. The fire in modern plants is removed from the retort by means of arches, and arrangement is made for circulation of the furnace gases around the retort in such a way as to secure the most effective

utilization of the heat. The continuous or mechanical retort consists of an oblong metal box divided horizontally across the middle by a plate; chain drums at each end, operated by gearing, serve to carry endless chains with a series of scrapers over the horizontal plate the entire length of the retort, returning along the bottom plate to the point of starting. The chips are fed into the retort at the same end as discharged, and thus travel twice the length of the retort before being completely carbonized and ready for removal. The gases leave from the top of the retort and pass to the condensers in the usual manner. Temperature control is of prime importance in its effect on the yields of products obtained. The best results are obtained by slow distillation during the critical stage of the reaction, whilst the exothermic reaction of the wood is taking place. This is accomplished by raising the temperature rapidly during the preliminary drying stage, and then reducing the amount of heat supplied at the commencement of the distilling stage, *i.e.* when the particles of tar first appear in the distillate. In this manner a smaller decomposition of the prime products takes place. The greatest increase in yield is wood alcohol, 30 per cent., and calcium acetate, 15 per cent.; when this mode of operation is employed. With the uncontrolled method the temperature in the retort flues ranges from 420° C. to 440° C. during the drying stages, and from 440° C. to 470° C. during the distillation stage, whilst with the improved method of firing the temperature ranges from 390° C. to 430° C. during the drying, and from 420° C. to 445° C. during distillation.

The immediate crude products are charcoal, non-condensable gas, tar, oils, and acid liquor. From the acid liquor calcium acetate is produced by neutralization with lime, distillation to remove wood spirit, and evaporation to dryness. The yield of acetic acid varies with the species of wood used, from 2·47 per cent. from soft pine, to 6·48 per cent. from birch, the average being 5·47 per cent. The influence of moisture on the yield of products in the destructive distillation of hardwood is set forth in the report of

Palmer and Cloukey, *Jour. Ind. Eng. Chem.*, 1915, 7. It was found that moisture has a favourable influence on the yield of acetic acid in controlled distillation. The effect of catalysts is also dealt with by Palmer, *Jour. Ind. Eng. Chem.*, 1918, 10, 264. Wood chips impregnated with aqueous phosphoric acid, and then distilled, gave largely increased yields of acetic acid. An increase from 5·13 per cent. tc 13·85 per cent. was obtained in one experiment, in which 2·45 per cent. of catalyst was added.

To obtain ordinary commercial acetic acid, acetate of lime is mixed with calcium chloride in proper proportions, and the solution concentrated until it crystallizes. The mother liquid is poured off and concentrated with the production of a second crop of crystals; this is repeated until about four crops have been obtained. The crystals are then dissolved in water, filtered through animal charcoal mixed with about 10 per cent. of calcium chloride and recrystallized. The crystals are distilled with a mixture of 1 part sulphuric acid of specific gravity 1·84 and 2 parts water, and the resulting acetic acid concentrated. This acid should contain 33 per cent. by weight of hydrogen acetate.

**Synthetic Acetic Acid.**—During the war large quantities of acetic acid have been manufactured synthetically; the process being the same as that described under synthetic alcohol in this volume, as far as the production of acetaldehyde. This substance is converted into acetic acid by oxygen obtained by the fractional distillation of liquid air in the presence of a catalyst. According to the Drefus patents (*e.g.* French Patent, No. 479656/1916, and British Patent, No. 105064/1917), which have been operated in the production of acetaldehyde and acetic acid from acetylene, the gas is passed with water: (*a*) into solvents in which mercury is soluble, *e.g.* sulphuric, phosphoric, and acetic acids; or (*b*) into solvents in which acetylene is soluble, *e.g.* acetone. In the former case, one or more of the following conditions are observed:—

(1) With sulphuric acid, a concentration limit of 5 to 20 per cent. is set.

(2) The absorbing solution contains less than 20 per cent. of the mercury compound.
(3) Throughout the absorption, the liquid is kept below 60° C.
(4) The acetylene is introduced in such quantity that it is all absorbed.
(5) The acetylene is at first introduced slowly until the mercury compound becomes grey or greyish-black.
(6) After a quantity of aldehyde has been formed the introduction of the gas is stopped, the temperature raised, aldehyde is distilled off, after which the temperature is again lowered and the process repeated.
(7) The gas employed is purified from sulphuretted hydrogen, phosphine, ammonia, and similar poisons.
(8) Water is added throughout the reaction in order to keep the acid concentration constant.
(9) The mixture of gas and liquid is strongly agitated to ensure intimacy of contact, the gas being under a slight positive pressure.

The process is carried out in an apparatus constructed of, or lined with lead, the surface of which is previously coated with a layer of lead sulphate or basic lead sulphate by treatment with sulphuric acid or mixtures of sulphuric and nitric acids.

The reaction mixture is cooled either externally or internally with cooling pipes protected against the action of acid. The acetic acid obtained by this process is of a high purity.

*Properties.*—Acetic acid as pyroligneous acid is used in calico-printing; in the preparation of varnishes and colouring matters; in the laboratory and certain industries as a solvent; in photography; in medicine as a local irritant, and to allay fever; and in the form of smelling salts.

For medicinal use 2 c.c. of acetic acid, specific gravity 1·044, diluted with 10 c.c. of water, should not immediately discharge the colour of one drop of solution of potassium permanganate. Also 5 c.c. should require for neutralization

28·7 c.c. of N. sodium hydroxide. There should be no residue on evaporation, and no characteristic reactions for copper, chlorides, nitrates, sulphates, and sulphites.

**Glacial Acetic Acid** is usually prepared by the distillation of a dry acetate with an equivalent quantity of strong sulphuric acid or acid potassium or sodium sulphate.

Sodium acetate is generally used. The anhydrous salt is fused on sheet-iron pans, the mass is cooled, broken into small lumps, and distilled with concentrated sulphuric acid. The first portion distilling contains the water, the latter portion is collected and cooled; when crystals have formed the still liquid portion is removed, the crystals are melted and redistilled as before, producing the glacial acid. 100 parts by weight should contain 98·9 parts of hydrogen acetate, the specific gravity of the liquid being 1·058, which rises on the addition of 10 per cent. of water. Each gramme diluted with 50 c.c. of water should require for neutralization 16·6 c.c. of N. sodium hydroxide.

**Aromatic Vinegar** is prepared by distilling crystallized diacetate of copper. The acetate is dried at 160° C. and heated in earthenware retorts, when the glacial acid distils over. The verdigris produces about half its weight of the acid. The pleasant odour is largely due to acetone, which is always produced when acetates of heavy metals are distilled. Other flavouring ingredients are, however, always added.

In the Acidum Aceticum Aromaticum of Belgium and Russia, oil of cloves, lavender, orange, bergamot; thyme, and cinnamon are used; while in the French preparation camphor is the chief flavouring ingredient.

## Section IV.—ACETONE AND GLYCERINE

**Acetone, $CO(CH_3)_2$,** is a mobile inflammable liquid, boiling at 56° C., with a specific gravity 0·8144 at 0° C. Before the war the main sources of acetone were the United States and Austria, in which countries it was produced from acetate of lime made by treating acetic acid with lime. The acetic acid is either obtained synthetically, as already described (p. 208), or by the destructive distillation of hardwood (p. 206). The vapours are passed into air-tight vessels, heated to about 500° C., containing porous material, such as pumice stone or bricks, the pores have been filled with the carbonate of an alkaline earth, such as lime or baryta. The porous material is packed into iron cylindrical retorts, which are heated to 500° C., and heated acetic vapour is introduced. This coming into contact with the large surface of the alkaline carbonate is decomposed into acetone, water, and carbon dioxide. The porous nature of the packing material obviates the necessity for mechanical agitation, which has been found necessary when the carbonates of the alkaline earths are employed in this method in any other form. When war broke out the demand for acetone was greatly increased. The quantity produced in this country was practically negligible as compared with the large and rapidly increasing requirements of this solvent for the manufacture of cordite, and later on for aeroplane dope.

In 1915 Dr. C. Weizmann brought to the notice of the Admiralty the production of acetone by bacterial fermentation from maize or other substances containing starch.

This process had previously been investigated by Fernbach as far back as 1910. He obtained acetone and butyl

alcohol from starchy raw material by fermentation, and a factory was started by the Synthetic Products Co. at King's Lynn. Potatoes were used as the raw material, and the bacteria were those of Prof. Fernbach.

Up to the time of the war, however, the butyl alcohol was considered of greater importance, and efforts were being made to utilize the product for the manufacture of synthetic rubber.

The failure of the Synthetic Products Co. to produce acetone in large quantities from potatoes resulted in the factory being taken over by the Ministry of Munitions, and at the same time other factories were fitted up, but grain was substituted for potatoes, and Weizmann bacteria used.

When grain became short and had to be reserved for food, resource was made to horse chestnuts with promising results.

The manufacture of acetone by the Weizmann process attained the greatest success at the factory of British Acetones, Toronto, Ltd., in Canada, where an output of nearly 200 long tons a month was reached.

In the fermentation of maize or other substances containing starch by this process, acetone and butyl alcohol are produced in the proportions of approximately 1 part of acetone to 2 of butyl alcohol.

As butyl alcohol had only a restricted use, both for war and industrial purposes, experiments were started by Dr. Weizmann in order to develop a process for converting normal butyl alcohol into methyl-ethyl ketone which, in the pure state, is equally suitable as acetone for the manufacture of cordite. The process, which was a catalytic one, was worked out on a laboratory scale, and promised to give good yields; it was decided to erect a large-scale plant at Toronto, which, however, only commenced successful operation just before the signing of the Armistice.

The results obtained during the war have proved that acetone can be produced successfully on a large manufacturing scale by the fermentation of substances containing starch. The process cannot, however, be considered as a

commercial one likely of being capable of competing with the production of acetone by the destructive distillation of wood, because of the relatively high cost of the raw material, and the fact that the production of every part of acetone involves the production of 2 parts of butyl alcohol, having very little value for industrial purposes, at all events, at the present time. From the purely practical point of view, the chief field for investigation is the utilization of the waste products. In a maize mash fermentation there is a solid residue of approximately 13 per cent. of the total maize used. This has a very high oil content, since during fermentation the maize oil is untouched. The albuminoid content is also fairly high, because all the nitrogen present is not used up by the bacilli during fermentation.

Another problem for the technical investigators is the utilization of the waste gases produced, the amount being approximately 5 cubic feet per lb. of maize used. Another point is the disposal of the butyl alcohol, which before the war was being experimented on for conversion into synthetic rubber.

In foreign countries the raw material can be grown cheaply and bought on the spot; for example, in India unpolished rice might be used. Millet is also available in large quantities.

It seems, therefore, that the future of the Weizmann process lies abroad, rather than at home, although from the pure research point of view much might be attained in the laboratories in this country.

**Weizmann Micro-organism.**—The micro-organism used for the production of acetone and butyl alcohol from cereals containing starch is a bacillus of the long rod type, which is stained readily with carbol fuchsin, but only lightly with methylene blue. In a vigorous culture spore formation will not take place much before the lapse of 20 to 24 hours. Growth occurs readily in various liquid media, that which is used most frequently being 5 per cent. maize mash. It is possible to rear colonies on a solid medium made by adding 2 per cent. agar to partially fermented maize mash.

A recipe used with excellent results is 1000 c.c. wort of specific gravity 1·008, 1 per cent. of gelatin, 1 per cent. calcium carbonate, and 2 per cent. agar. Heat gently, filter, sterilize, and make up into tubes. Attempts to prepare plate cultures with this medium have been unsuccessful.

Since acetone is inflammable and has a low boiling-point and gravity similar to petrol, it works satisfactorily in an internal combustion engine, and could, therefore, if produced economically, supplement the latter during the present shortage.

**Glycerine from Molasses.**—Pasteur in 1858 observed that glycerin, and succinic acid, in traces are products of the so-called alcoholic fermentation of the sugars. There is every reason to believe that the glycerine formed in this way owes its origin directly to the sugars, and not to the secondary constituents always present in those fundamental liquids, worts, must, etc., met with in commerce. Despite numerous attempts to obtain glycerine in quantities which would be commercially profitable, up to quite recently no success has been met with.

In the summer of 1917 it was known that the Germans were producing glycerine in large quantities by the fermentation of sugars.

John R. Eoff three months later was able to produce glycerine in such quantities that if the actual cost of the recovery was not too high, the process would be commercially profitable.

The following is an outline of his experiments: The sugars were fermented with 5 per cent. of sodium carbonate, which must not be added to the liquid all at once. A less quantity of the alkali diminishes the yield of glycerine, whilst a larger quantity stops fermentation; other alkaline substances may be used, but sodium carbonate is preferable on account of its cheapness. It should be added as soon as the fermentation has well started, and in as large quantities and as frequently as is possible without stopping fermentation. The earlier the addition of the alkali, the higher the

yield of glycerine will be. Pure yeast cultures are most suitable, and the presence of ammonium chloride in the fermenting liquid augments the yield of glycerine. The most favourable temperature for the fermentation is 30° to 32° C., higher temperatures lead to a loss of alcohol and glycerine and to the formation of objectionable substances, whilst smaller yields of glycerine are obtained at lower temperatures. The most favourable concentration for the sugar solutions lie between 17·5 and 20 grams of sugar per 100 c.c. When fermentation is complete, according to this method, 20 to 25 per cent. of the sugar originally present in the liquid is converted into glycerine, and practically all the remainder into alcohol and carbon dioxide.

When the sodium carbonate has been added to the fermenting solution in sufficient quantity, a copious precipitate is formed, the evolution of gas ceases, and the yeast apparently lies dormant for awhile. The precipitate eventually disappears, and the fermentation again proceeds. It is essential that the above reactions should take place. Solid sodium carbonate produces better results than if it is added in the form of a solution. The purification of the fermented wash is carried out as follows: The wash is neutralized with sulphuric acid, and a saturated solution of commercial ferrous sulphate (copperas) added. The wash is then nearly brought to the boil, milk of lime added, until there is excess of lime in solution, when the wash is boiled for half an hour. The liquid is then passed through a filter press and the cake steamed. The lime and copperas treatment is then repeated, and after again being passed through a filter press the alkalinity is brought to 0·2 per cent. ($Na_2CO_3$) by the addition of soda ash. It is then filtered, pressed, and steamed, and the filtrate evaporated to a thick syrup. It is then distilled in a still resembling that of Jobbin, only about half the glycerine present in the fermented wash being obtained.

Experiments have been carried out on a commercial scale, using inedible "black strap" Porto Rico molasses; as only half the glycerine present in the fermented wash is

recovered as crude glycerine, the yield works out therefore at about 5½ to 6 lbs. per cwt. of molasses dealt with.

However, the value of the alcohol at the present price should balance the cost of all material and overhead charges entering into the production of the fermented wash; so that the only cost to be considered for the glycerine would be that of purification and distillation.

Experiments have also been carried out with cane sugar and starch glucose as fermentable material. It was found necessary in these cases to employ yeast foods that deleteriously influenced the purification of the glycerine. It was therefore concluded that these materials had no superiority over molasses for the purpose.

As molasses in Australia and Fiji are waste products and run into the sea, the present process should be of greater value in such countries.

In Austria during the war large quantities of glycerine were made by the Connstein-Lüdecke process. In this process the fermentation proceeds in presence of sodium sulphite, acetaldehyde is produced in equivalent amount to the glycerine with a maximum yield of 38 per cent.

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# INDEX


ACETATES, 202
Acetic acid, 206
  synthetic, 208
  properties, 209
  glacial, 210
Acetification, 203
Acetone, 211
Acetylene, alcohol from, 197
Affination, 128
Alcohol, absolute, 176
  physiological action of, 184
  as a food, 184
  mental effects, 185
  action on the digestion, respiration, 186
  poisonous action, 188
  industrial, 190
  denaturants, 191
  synthetic, 194
Alcoholic fermentation, 140
Amylo fermentation process, 173
Animal charcoal, 129
Argol, 168
Aromatic vinegar, 210

*Bacterium xylinum*, 200
Bagasse, 61
Barium sucrate, 13
Barley, 140
Beet juice extraction, 94
  purification, 100
Beetroot, 83
  cultivation, 84
  selection, 85
  composition, 86
  washing, 94
  slicing, 94
  pulp, 99
Beet sugar manufacture, 87
  history, 88
Black strap, 215
Blankit, 65
Boiling beet juice, 113
  to grain, 116, 130
  control of, 117
Bolvin and Loiseau, 103
Brandy, 178
  British, 179
  cognac, 179
  "marc," 179
British wines, 161

CALCIUM sucrates, 13, 123
Cane mills, 54
  sugar, 45
  history, 45
  production, 48
  plant, 48
Caramel, 138
  properties, 138
  manufacture, 138
  uses, 139
Carbon, alcohol from, 196
Carbonatation, 69
Carbon dioxide, 108
Cellulose, 1
Centrifugals, 80
Char, 129
Clarification of cane juice, 64
  continuous, 66
Claassen's method of boiling, 117
Cognac, 179
Cooling and refrigerating, 149
Copper wall, 71
Cordials, 184
Crystallization, 77
  in motion, 120
Crystallizers, 79
Cube sugar, 131
Curin's method of boiling, 117
Curing, 78
Cush-cush, 63

DEFECATION, 101
  single and double, 102
  agents, 64
Defecators, 66
Demerara rum, 180
Deming's superheat clarification, 6
Denaturants of alcohol, 191
Destructive distillation, wood, 195
  peat, 196

Dextrin, 5, 31
torrefaction process, 31
acid process, 32
uses of, 33
references, 43
Diastase, 142
Diffusion battery, 95
Hyross Rak, 98
process, 57
beet, 92
theory of, 93
Distillation, 174
Drying massecuite, 130
Dubrunfaut, 122
and Leplay, 123

ELIMINATORS, 68
Elution process, 123
Ethylene, alcohol from, 198
Evaporation, 71
beet juice, 109
Ewen-Tomlinson process, 195
Exhausted pulp, 99
drying, 99

FECULOSE, 33
Feints, 181
Fermentation, 151
changes during, 153
Fernbach, acetone process, 121, 211
Filtering media, 71
Filter press, 106
Filtration of cane juice, 128
through char, 129
of beet scums, 105
Fore shots, 181
Formaldehyde, 65
Fructose, 9
preparation, 10
properties, 10
Fryer's concretor, 72

GALACTOSE, 11
Geneva, 183
Gin, 182
Glacial acetic acid, 210
Gloy, 33
Glucose, 7, 34
properties, 8
manufacture, 36
solid, 39
references, 43
Gluten, 20
Glycerine from molasses, 214
Grain spirit, 170
malting and mashing, 170
Granulated sugar, 131
Grape sugar, 39
references, 44
Grist, 143

HATTON'S continuous defecator, 66
Herzfeld, solubility of sugar, 114
Hollands, 183
Hops, 148
Hydro-sucro carbonate of lime, 103
Hydrosulphites, 65
Hyross Rak's process, 98

INDUSTRIAL alcohol, 190
Invertase, 10
Invert sugar, 10
properties, 11
preparation, 132
Irish whiskey, 181

JAMAICA rum, 179

KESSELER'S extraction process, 61
Kestner evaporator, 75

LACTOSE, 14
properties, 14
Lees, 168
Lillie evaporator, 75
Lime kilns, 107
Ling, glycerine process, 122
Liqueurs, 184

MACERATION and imbibition, 55
of beetroots, 90
Maize or corn starch, 20
Malting, 141
Maltose, properties, 13
preparation, 42
references, 44
Maple sugar and syrup, 135
Marc, 164
Mashing, changes during, 145
Massecuite, 118
treatment, 119
Megass, 61
Melting, 128
Molasses, beet, 121
acetone from, 121
glycerine from, 122, 214
fodder, 100
composition, 80
utilization, 81
Multiple effect, 112
crushing, 56
Muscovado sugar, 77
Must, 163
constituents of, 164
*Mycoderma aceti*, 200
*vini*, 200

NAUDET'S process, cane, 60
beet, 97

OSMOSE process, 122

PALM sugar, 136
Panela train, 72
Patent stills, 174
Peat, alcohol from, 196
Potato, 183
  starch, 24
  rasper, 27
Pot still, 174
Pressing beetroots, 89
Proof spirit, 176

RAFFINOSE, 14
  properties, 14
Raw sugars, 127
Recovery of sugar from molasses, 122
Rectifying, 175
Refinery molasses, 132
Refining, 127
Rice starch, 22
Rum, 179

*Saccharomyces ellipsoideus*, 165
Saccharum, 11
  preparation, 132
Sago, 29
Saturation, 117
Scheibler, 123
Schnapps, 183
Scotch whiskey, 181
Scums, 107
  treatment of, 70
Separation process, 123
Shoemakers' paste, 33
Sorghum sugar and syrup, 134
Spirit, proof, 176
  potable, 178
  grain, 170
  potato, 183
  grape, 184
*Spiritus rectificatus*, 175
Starch, 3
  arrowroot, 29
  fermentation process, 18
  maize, 20
  potato, 24
  properties, 4
  references, 43
  rice, 22
  soluble, 29
  sources, 16
  statistics, 40
  sweet process, 19
  uses, 30
Steffen, 123
Stills, patent, 174
Stills, pot, 174
  Coffey, 174
Strontia process, 123
Strontium sucrate, 13
  process, 124
Substitution process, 123
Sucrose, 11, 45
  properties, 45
Sugars, 6
Sugar substitutes, 7
Sugar cane, 48
  varieties, 52
  seedlings, 52
  pests and diseases, 52
  juice extraction, 53
  yields, 62
  juice, 63
Sugar beet, 83
  production, 125
Sulphiting, 105
Sulphur burner, 109
Sulphuring, 68
Supersaturation, 117

TABLE syrups, 132
Tapioca, 29
Tartar, 168
Tartaric acid, 168
Triple effect, 73

VACUUM pan, 73, 78
  beet, 115
Vinegar, wort, 200
  quick process, 201
  malt, 203
  wine, 203
  from alcohol, 204
  aromatic, 210

WASTE sulphite liquor, 195
Water for brewing, 147
Weizmann, 211
Wheat grain, 17
Whiskey, 181
Wine, 161
  must, 163
  slow fermentation, 165
  storage, 166
  lees, 168
Wines, sweet, dry, effervescing, and medicated, 166
Wort, 145, 148

YEAST, 152

ZYMASE, 159

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BAILLIÈRE, TINDALL AND COX
8, HENRIETTA STREET, COVENT GARDEN, LONDON, W.C. 2